UNIVERSITAT POLITÈCNICA DE CATALUNYA
BARCELONATECH
Escola d'Enginyeria de Barcelona Est

BACHELOR'S THESIS

Bachelor of Mechanical Engineering

HOT DEFORMATION CHARACTERISATION OF HAYNES-242

Report and Annexes

Author: Gaurav Venkatesha Babu
Director: Jessica Calvo

Date: June 2017

ABSTRACT

Ni-based superalloys are metallic materials with exceptional combinations of creep and oxidation resistance at high temperatures. Wrought alloys are hot formed at high temperatures and subsequently annealed and aged. The final mechanical properties of the alloys are a combination of the grain size generated during the process and the precipitation state after aging. In fact, precipitation phenomena are complex for these alloys, which contain up to 12 different elements in their compositions. This study will deal with the forming characteristics of Haynes 242. Prior to forming, usually by forging, the material has to be reheated and this initial thermal treatment can affect grain size. The reheating stage is defined by the temperature and soaking time at the selected temperature. The best combination of these two parameters has to warranty that forging will be performed above the solvus temperature of precipitates without promoting excessive grain growth. The initial part of the study will analyse the effect of different temperatures and soaking times on the grain size and precipitates. After reheating, forging operations are performed at different forming temperatures and strain rates. Forging conditions will be simulated by means of hot compression testing. Compression samples will be first reheated according to the optimal conditions defined in the first part of the study, and then compressed at different temperatures and strain rates. Hot flow curves will be obtained which will be used for the analysis of the hot flow behaviour of the alloy. Metallographic evaluation of the compressed samples will assess the evolution of the grain size during forming and the mechanisms taking place, i.e. dynamic recrystallization or dynamic recovery, according to the deformation parameters. These results will be useful for the assessment of hot deformation operations (reheating and forging conditions) for Haynes 242, leading to fine and homogeneous microstructures.

ACKNOWLEDGEMENTS

I would first like to thank my thesis advisor Dr. Jessica Calvo of the PROCOMAME research group at UPC. The door to Prof. Jessica Calvo office was always open whenever I ran into a trouble spot or had a question about my research or writing.She consistently allowed this thesis to be my own work, but steered me in the right the direction whenever he thought I needed it.

I place on record, my sincere thank you Dr.Jose Maria Cabrera for accepting my request to do my bachelor thesis under his esteemed research group.I am also very thankful to Dr. Nagaraja, HOD of Mechanical Engineering Dept, ASE-B for his continuous support.

I take this opportunity to express gratitude to Mr.Pramod, Mr. Phanibhushan and other Mechanical Department faculty members at Amrita University for their help and constantly bolstering through thick and thin of my engineering life.

I am also grateful to Dr Pedro Paramo and Mr Utku Deniz. I am extremly thankful and indebted to them for sharing expertise, sincere and valuable guidance and encouragement extended to me.

I need to thank all who create such good atmosphere in the lab; Cynthia, Marcelo Caprio, Ana Isabel Macias.

Last but not the least, I would like to thank my parents for allowing me to realize my own potential. All the support they have provided me over the years was the greatest gift anyone has ever given me and for teaching me the value of hardwork and an education. Without them, I may never have gotten to where I am today.

INDEX

Contents

1. INTRODUCTION TO HAYNES 242

1.1 PRINCIPAL FEATURES

High-Temperature Strength, Low Thermal Expansion Characteristics, and Good Oxidation Resistance

HAYNES 242 alloy (UNS N10242) is an age-hardenable nickel-molybdenum chromium alloy which derives its strength from a long-range ordering reaction upon aging. It has tensile and creep strength properties up to 1300°F (705°C) which are as much as double those for solid solution strengthened alloys, but with high ductility in the aged condition. The thermal expansion characteristics of 242 alloy are much lower than those for most other alloys, and it has very good oxidation resistance up to 1500°F (815°C). Other attractive features include excellent low cycle fatigue properties, very good thermal stability, and resistance to high-temperature fluorine and fluoride environments [1].

Fabrication

HAYNES® 242® alloy has very good forming and welding characteristics in the annealed condition. It may be forged or otherwise hot-worked by conventional techniques, and it is readily cold formable. Welding may be performed in the annealed condition by standard gas tungsten arc (GTAW) or gas metal arc (GMAW) techniques. Use of matching composition filler metal is suggested. For further information on forming and fabrication, contact Haynes International. [1]

Heat-Treatment

HAYNES® 242® alloy is furnished in the annealed condition, unless otherwise specified. The alloy is usually annealed in the range of 1900-2050°F (925-1120°C), depending upon specific requirements, followed by an air cool (or more rapid cooling) before aging. A water quench is recommended for heavy section components. Aging is performed at 1200°F (650°C) for a period of 24-48 hours, followed by an air cool. HAYNES® 242® alloy is produced in the form of reforge billet, bar, plate, sheet, and wire welding products, all in various sizes. Other forms may be produced upon request.[1]

Applications

HAYNES® 242® alloy combines properties which make it ideally suited for a variety of component applications in the aerospace industry. It will be used for seal rings, containment rings, duct segments, casings, fasteners, rocket nozzles, pumps, and many others. In the chemical process industry, 242® alloy will find use in high-temperature hydrofluoric acid vapor-containing processes as a consequence of its excellent resistance to that environment. The alloy also displays excellent resistance to high-temperature fluoride salt mixtures. The high strength and fluorine environment-resistance of 242® alloy has also been shown to provide for excellent service in fluoroelastomer process equipment, such as extrusion screws. HAYNES® 242® alloy derives its age-hardened strength from a unique long-range-ordering reaction which essentially doubles the un-aged strength while preserving excellent ductility. The ordered Ni2 (Mo,Cr)-type domains are less than a few hundred Angstroms in size, and are visible only with the use of electron microscopy[22]

Nominal Composition

Typical composition of H-242 is given in the table 1.

Table 1 chemical composition of H-242(%weight)[1]

Nickel	Bal
Molybdenum	25
Chromium	8
Iron	2 max
Cobalt	1 max
Manganese	0.8 max
Silicon	0.8 max
Aluminum	0.5 max
Carbon	0.03 max
Boron	0.006 max

1.2 STRESS RUPTURE STRENGTH

HAYNES® 242® alloy is an is an age-hardenable material which combines excellent strength and ductility in the aged condition with good fabricability in the annealed condition. It is particularly effective for strength-limited applications up to 1300°F (705°C), where its strength is as much as double that for typical solid-solution strengthened alloys. It may be used at higher temperatures, where its solid-solution strength is still excellent, but oxidation resistance limits such uses to about 1500-1600°F (815-870°C).[1]

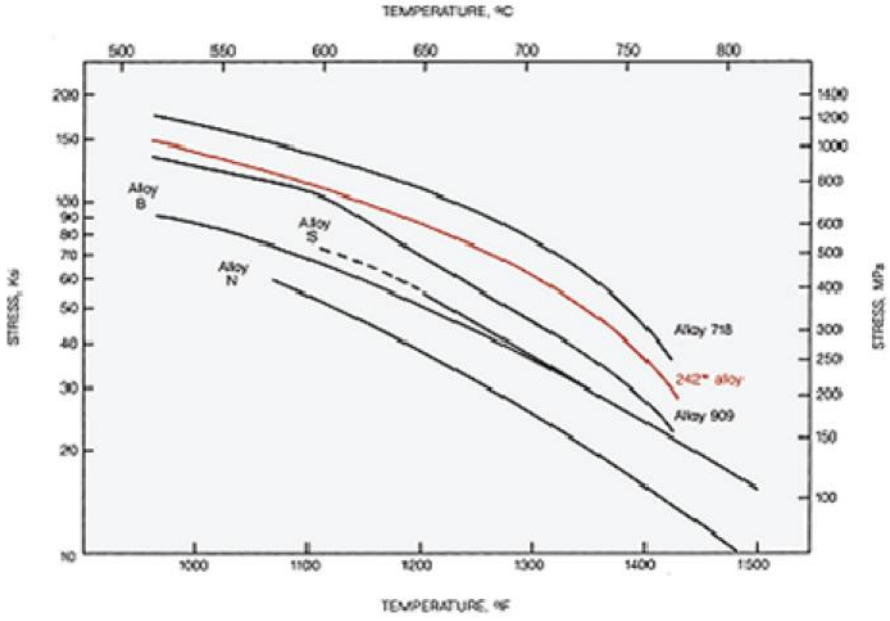


Fig 1.2 Comparison of 100 Hour Stress-Rupture Strengths [1]

1.3 COMPARISIONS OF YIELD STRENGTH AND ELONGATIONS

HAYNES 242® alloy exhibits much higher yield strength than typical solid-solution- strengthened nickel-base alloys, such as HASTELLOY® S alloy, but also possesses excel- lent ductility in the fully heat treated condition. This can translate into excellent containment characteristics for gas turbine rings and casings, particularly when coupled with 242® al- loy's lower expansion coefficient and excellent ductility retention following thermal expo- sure. This combination is also well suited for a range of fastener and bolting applications up to 1300°F (705°C).[1]

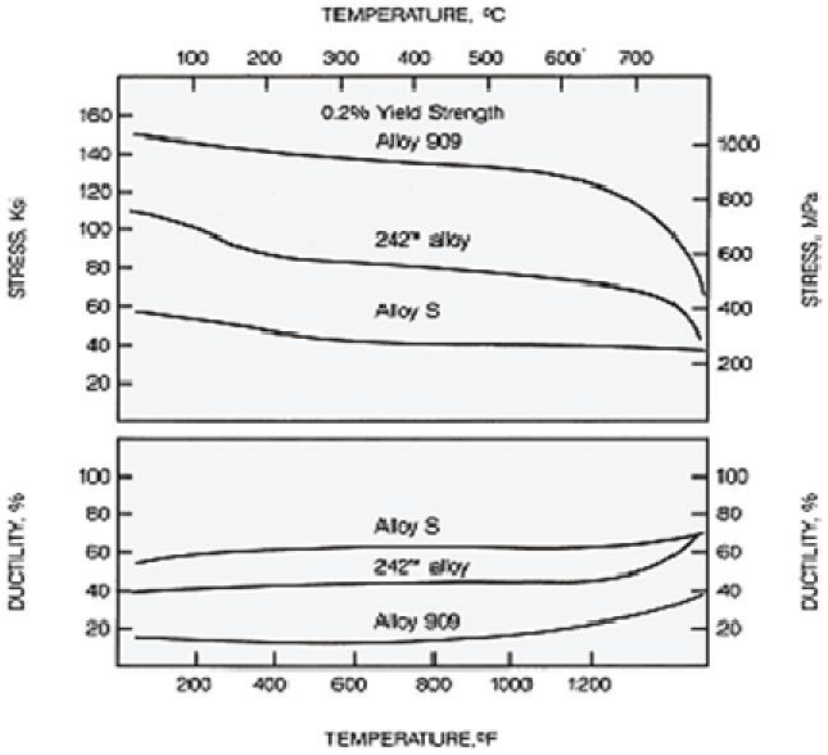


Fig 1.3 yield stress vs temperature[1]

1.4 COMPARISION OF THERMAL EXPANSION CHARACTERISTICS

HAYNES 242 alloy exhibits significantly lower thermal expansion characteristics than most nickel-base high-temperature alloys in the range of temperature from room tempera-ture to 1600°F (870°C). Although its expansion is greater than that for alloy 909 below 1000°F (540°C), at higher temperatures, the difference narrows considerably.[1]

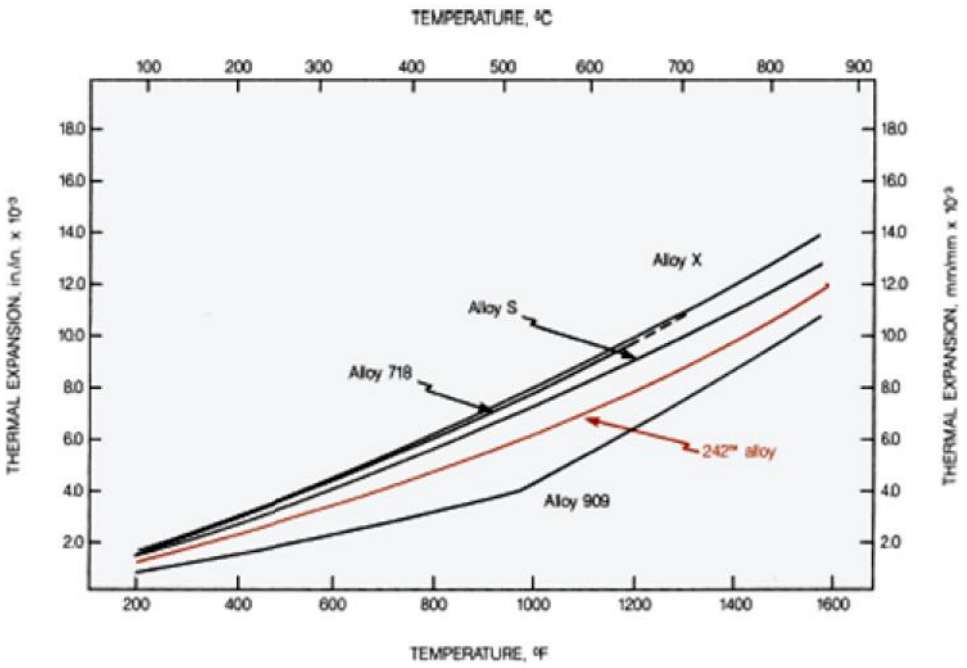


Fig 1.4. Total Thermal Expansion, Room to Elevated Temperature[1]

1.5 THERMAL STABILITY

HAYNES 242 alloy has excellent retained ductility and impact strength after long-term thermal exposure at temperature. Combined with its high strength and low thermal expansion characteristics, this makes for very good containment properties in gas turbine static structures. The graphs below show the retained room-temperature tensile elongation and impact strength for 242 alloy versus other relevant materials after a 4000 hour exposure at 1200°F (650°C).[1]

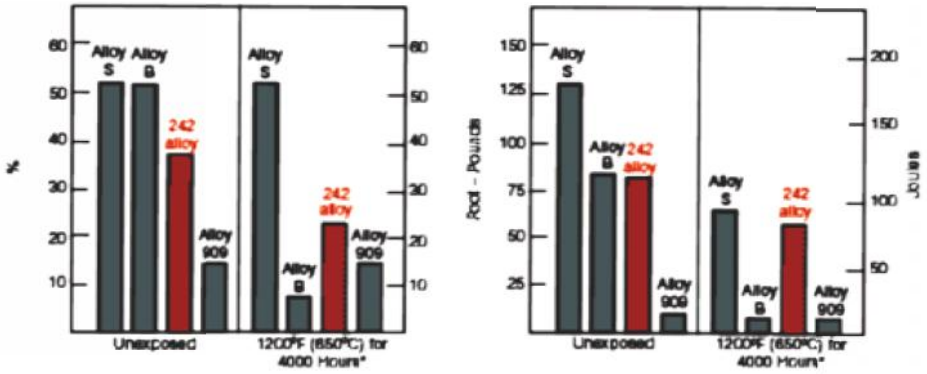


Fig 1.5.a) Comparative Retained Ductility and b) Impact Strength Room-Temperature TensileElongation Room Temperature Impact Strength[1]

1.6 WELDING

HAYNES 242 alloy has excellent forming and welding characteristics(Fig 1.6a). It may be hotworked at temperatures in the range of about 1800-2250°F (980-1230°C) provided the entire piece is soaked for a time sufficient to bring it uniformly to temperature. Initial breakdown is normally performed at the higher end of the range, while finishing is usually done at the lower temperatures to afford grain refinement. As a consequence of its good ductility, 242 alloy is also readily formed by cold-working. All hot or cold-worked parts should be annealed at 1900-2050°F (925-1120°C) and cooled by air cool or faster rate before aging at 1200°F (650°C) in order to develop the best balance of properties. The alloy can be welded by a variety of processes, including gas tungsten arc, gas metal arc, and shielded metal arc. High heat input processes such as submerged arc and oxyacetalyne welding are not recommended.[1]

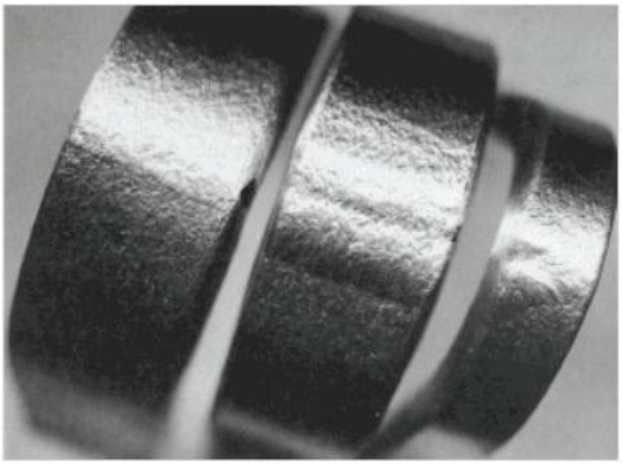


Fig 1.6 Typical root, face, and side bends (L to R) for welded 242 alloy[1]

2. OBJECTIVE

High temperature forming is widely used for the fabrication of metallic materials such as superalloys. At high temperatures, the amount of deformation which can be applied is higher than at lower temperatures and the stresses are significantly lower. Additionally, the material becomes more dynamic in nature. At high temperatures, mechanisms such as dynamic or metadynamic recrystallization and recovery can take place, offering an opportunity to control the microstructural features, being grain size the most important one in terms of its effect on the mechanical properties. In this project, Haynes 242 will be deformed at different temperatures and strain rates. The effect of these parameters on the stress strain flow curves will be analysed, as well as the evolution of the microstructure in terms of the recrystallized fraction and recrystallized grain size.

3. EXPERIMENTAL SETUP

3.1 HEAT TREATMENT

3.1.1 SOLUTI0NIZING TREATMENT

Heat treating is a group of industrial and metalworking process used to alter the physical, and sometimes chemical, properties of a material. The most common application is metallurgical. Heat treatments are also used in the manufacture of many other materials, such as glass. Heat treatment involves the use of heating or chilling, normally to extreme temperatures, to achieve a desired result such as hardening or softening of a material. Heat treatment techniques include annealing, case hardening, precipitation hardening, tempering, normalizing and quenching. It is noteworthy that while the term *heat treatment* applies only to processes where the heating and cooling are done for the specific purpose of altering properties intentionally, heating and cooling often occur incidentally during other manufacturing processes such as hot forming or welding.[4]

Solutionizing treatment is done in order to dissolve the precipitates and there by forming homogenous solid solution.[5] This treatment is carried at 1066°C for about 30 minutes(fig 3.1.1b) in 'HOBERSAL tubular furnace'(Fig 3.1.1a) and then followed by water quenching. In materials science, quenching is the rapid cooling of a work piece to obtain certain material properties. A type of heat treating, quenching prevents undesired low-temperature processes, such as phase transformations, from occurring. Arrange the specimen in a ceramic tray with all protective gears on. Open the lid of the furnace and place the specimen tray at the middle of the tubular furnace and Close the furnace; make sure that it's tightened moderately. Choose the respective program and set open the argon gas valve. After 30 minutes take out the specimens and quench it (water quenching) and follow specimen preparations that are discussed in section 3.3.

Fig 3.1.1a Hobersal Tubular furnace

Fig 3.1.1b specimen ready to be quenched(1066˚C)

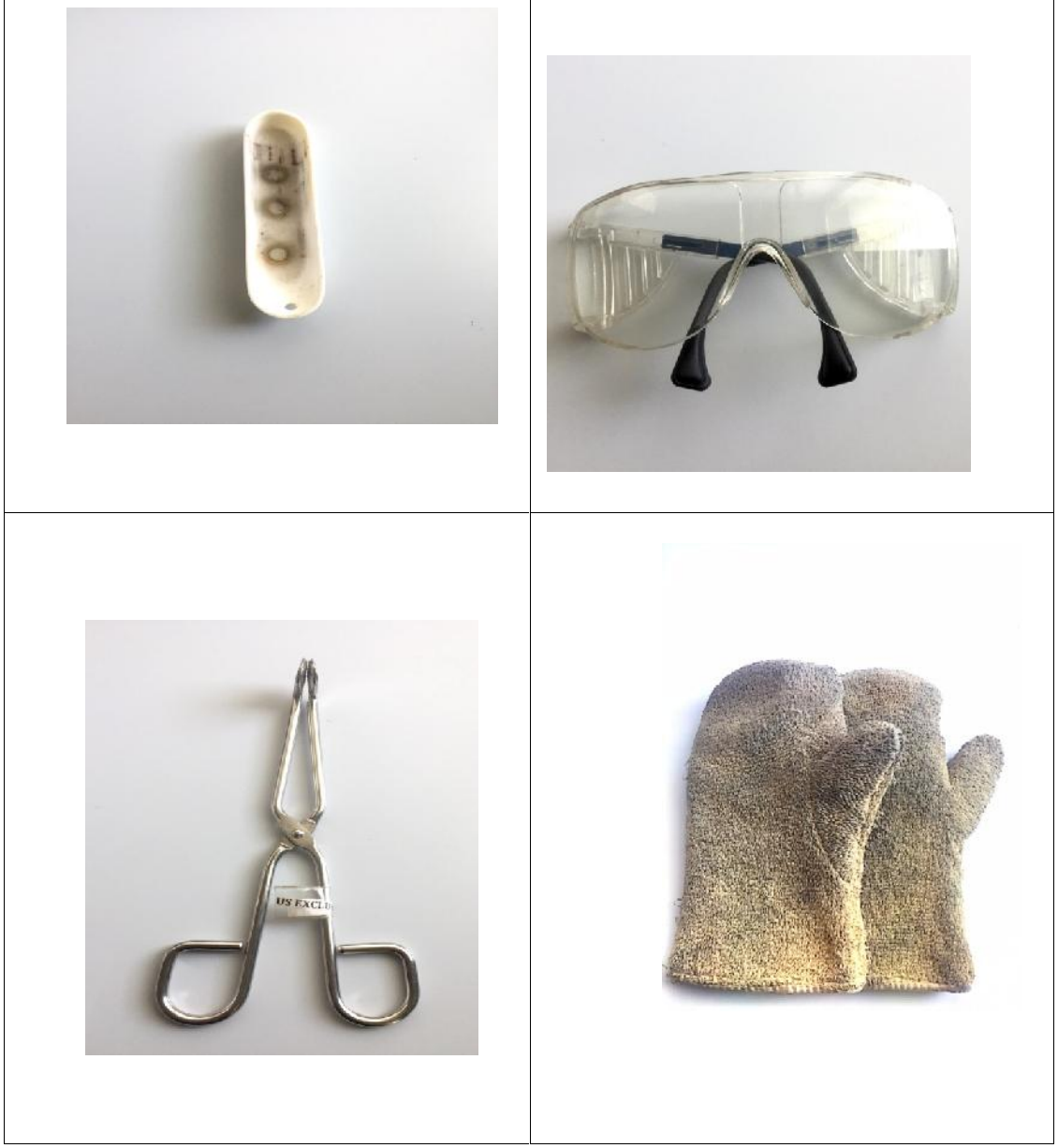


Fig 3.1.1c Preventive and necessary items required during heat treatment process

(Clockwise) specimen holder, safety goggles, lab tongs, safety gloves

3.1.2 AGING TREATMENT

Age hardening is a heat treatment technique used to increase the yield strength of malleable materials, including most structural alloys of aluminum, magnesium, nickel, titanium, and some steels and stainless steels. In superalloys, it is known to cause yield strength anomaly providing excellent high-temperature strength.[5]

Precipitation hardening relies on changes in solid solubility with temperature to produce fine particles of an impurity phase, which impede the movement of dislocations, or defects in a crystal's lattice. Since dislocations are often the dominant carriers of plasticity, this serves to harden the material. The impurities play the same role as the particle substances in particle reinforced composite materials. Just as the formation of ice in air can produce clouds, snow, or hail, depending upon the thermal history of a given portion of the atmosphere, precipitation in solids can produce many different sizes of particles, which have radically different properties. Unlike ordinary tempering, alloys must be kept at elevated temperature for hours to allow precipitation to take place. This time delay is called "aging".[5]

Note that two different heat treatments involving precipitates can alter the strength of a material: solution heat treating and precipitation heat treating. Solid Solution Strengthening involves formation of a single-phase solid solution via quenching. Precipitation heat treating involves the addition of impurity particles to increase a material's strength. During aging process precipitates starts to form as finely distributed particles and after this process hardness of the material is bound to increase. Aging process is carried out at 650°C for 24H in Hobersal tubular furnace(Fig3.1.2a). Arrange the specimen in a ceramic tray with all protective gears on.(Fig 3.1.2b) Open the lid of the furnace and place the specimen tray at the middle of the tubular furnace and Close the furnace; make sure that it’s tightened moderately. Choose the respective program and set open the argon gas valve. After 30 minutes take out the specimens and quench it (water quenching) and follow specimen preparations that are discussed in section 3.3.

Fig 3.1.2a Hobersal Tubular furnace

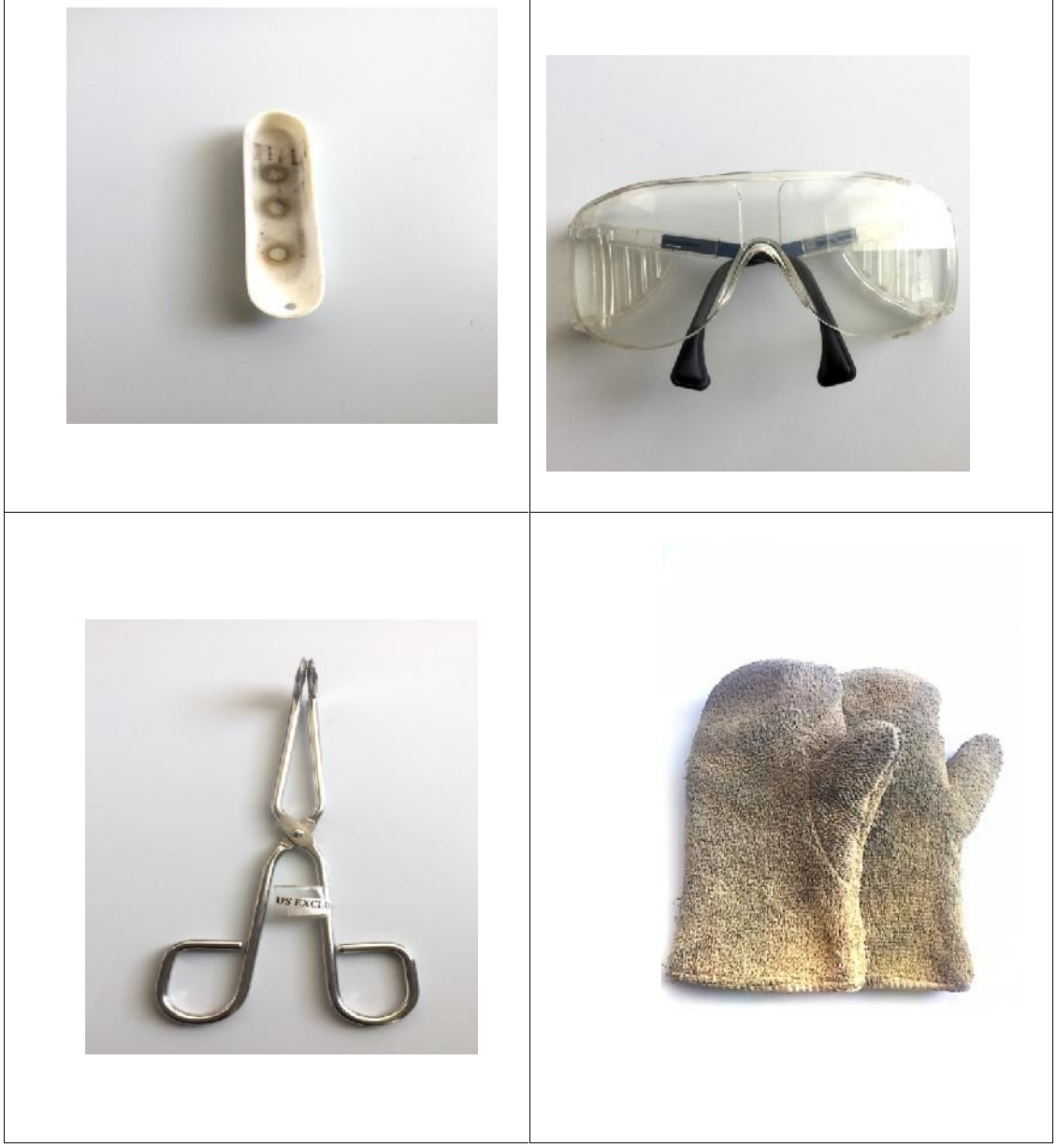


Fig 3.1.2b Preventive and necessary items required during heat treatment process

(Clockwise) specimen holder, safety goggles, lab tongs, safety gloves

3.1.3 QUENCHING TREATMENT

Although there is evidence of the use of quenching processes by blacksmiths stretching back into the middle of the Iron Age, little detailed information exists related to the development of these techniques and the procedures employed by early smiths. Although early ironworkers must swiftly have noticed that processes of cooling could affect the strength and brittleness of iron, and it can be claimed that heat-treatment of steel was known in the Old World from the late second millennium BCE, it is hard to identify deliberate uses of quenching archaeologically. Moreover, it appears that, at least in Europe, 'quenching and tempering separately do not seem to have become common until the 15th century'; it is therefore helpful to distinguish between 'full quenching' of steel, where the quenching is so rapid that only martensite forms, and 'slack quenching', where the quenching is slower or interrupted, which also allows pearlite to form and results in a less brittle product.[5]

Haynes 242 is heated up to the suitable temperature and then quenched in water to harden to it. Haynes 242 is heated to the suitable temperature for hardening, then cooled rapidly by immersing the hot part in water to transform the material to a fully hardened structure. (Fig 3.1.3a) Parts which are quenched usually must be aged, tempered or stress relieved to achieve the proper toughness, final hardness and dimensional stability

Fig 3.1.3a Water Quenching

3.2 COMPRESSIVE STRENGTH TESTING (INSTRON)

STRESS

The term stress (s) is used to express the loading in terms of force applied to a certain cross-sectional area of an object. From the perspective of loading, stress is the applied force or system of forces that tends to deform a body. From the perspective of what is happening within a material, stress is the internal distribution of forces within a body that balance and react to the loads applied to it.[12](Fig 3.2a)

STRAIN

Strain is the response of a system to an applied stress. When a material is loaded with a force, it produces a stress, which then causes a material to deform. Engineering strain is defined as the amount of deformation in the direction of the applied force divided by the initial length of the material [13]. This results in a unit less number. (Fig 3.2a)

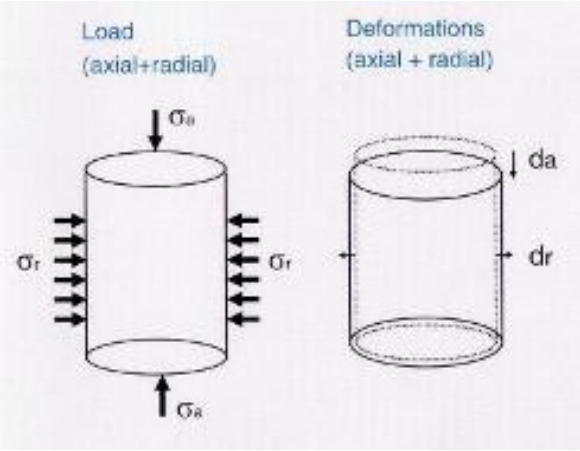


Fig 3.2a Schematic representation of stress and strain[17]

POISSON'S RATIO

Poisson's ratio denoted nu and named after Siméon Poisson, is the signed ratio of transverse strain to axial strain. Nu is the amount of transversal expansion divided by the amount of axial compression, for small values of these changes. Poisson's ratio is a measure of the Poisson effect, the phenomenon in which a material tends to expand in directions perpendicular to the direction of compression.[18](Fig 3.2b)

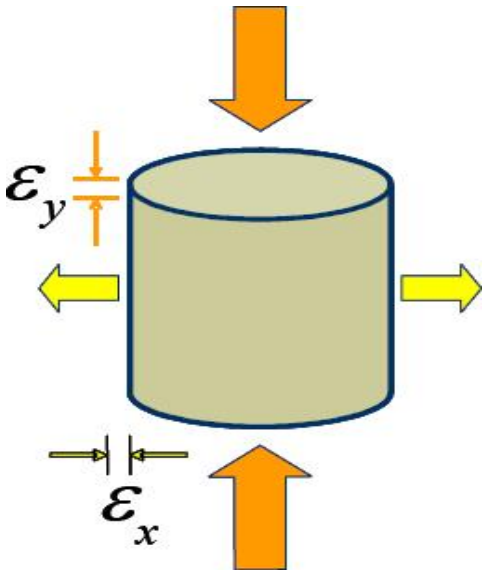


Fig 3.2b schematic representation of Poisson's ratio[19]

COMPRESSION TEST

Axial compression testing is a useful procedure for measuring the plastic flow behavior and ductile fracture limits of a material. Measuring the plastic flow behavior requires frictionless (homogenous compression) test conditions, while measuring ductile fracture limits takes advantage of the barrel formation and controlled stress and strain conditions at the equator of the barreled surface when compression is carried out with friction. Variations of the strains during a compression testAxial compression testing are also useful for measurement of elastic and compressive fracture properties of brittle materials or low-ductility materials. In any case, the use of specimens having large L/D ratios should be avoided to prevent buckling and shearing modes of deformation." (Fig 3.2c)(Fig 3.2d)[15]

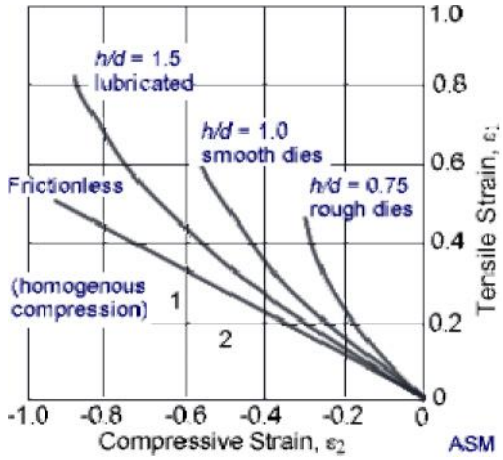


Fig 3.2c The image below shows variation of the strains during a compression test without friction (homogenous compression) and with progressively higher levels of friction and decreasing aspect ratio L/D (shown as h/d).[16]

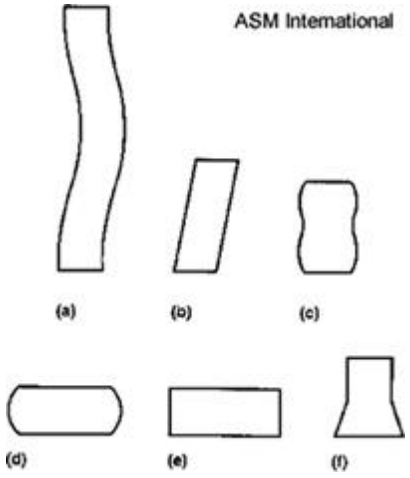


Fig 3.2d (a) Buckling, when L/D > 5. (b) Shearing, when L/D > 2.5. (c) Double barreling, when L/D > 2.0 and friction is present at the contact surfaces. (d) Barreling, when L/D < 2.0 and friction is present at the contact surfaces. (e) Homogenous compression, when L/D < 2.0 and no friction is present at the contact surfaces. (f) Compressive instability due to work-softening material.[16]

Cylindrical specimens, 11.4 mm in height and 7.6 mm in diameter, were prepared for hot compression testing using the Instron 4507 universal deformation machine. Tantalum foils and boron nitride solution were used to reduce friction in this case.[14] Single-hit hot compression tests were carried out at temperatures of 900–1050 C with strain rates of 0.001-0.1.

3.3 SPECIMEN PREPARATION FOR METALLOGRAPHY

3.3.1 CUTTING

Most metallographic samples need to be sectioned to the area of interest and for easier handling. Depending upon the material, the sectioning operation can be obtained by abrasive cutting (metals and metal matrix composites), diamond wafer cutting (ceramics, electronics, biomaterials, minerals), or thin sectioning with a microtome (plastics). Proper sectioning is required to minimize damage, which may alter the microstructure and produce false metallographic characterization.

The as received cylindrical shaped specimen is cut into 2 smaller sized specimen using ACCUTOM-50(Fig 3.3.1b).Mount the specimen on to the cutting machine using suitable gigs and fixtures and then choose the particular cutting disc and mount it on to the spindle and check for its tightness(Fig 3.3.1a). Fix the initial values of X and Y co ordinates and teach the machine as to where the machine has to stop its cutting and note down that relative position. Set the value of stop position of the mount carrying specimen and make sure that the stop position is always greater than the relative position. Press OK and wait till the process is finished.

Fig 3.3.1a The cutting disc

Fig 3.3.1b ACCUTOM-50, machine used for specimen cutting

3.3.2 HOT MOULDING PROCESS

Hot pressing is a high-pressure, low-strain-rate powder metallurgy process for forming of a powder or powder compact at a temperature high enough to induce sintering and creep processes. This is achieved by the simultaneous application of heat and pressure. With direct hot pressing technology, materials can be sintered to their final density. The near net-shape precision achieved is very high and often eliminates mechanical reworking of materials that are often difficult to process. In the friction material industry, direct hot pressing plays an increasing role in production of sintered brake pads and clutches. Sintered brake pads are increasingly used for high speed train and motorcycle applications, as well as wind energy, ATVs, mountain bikes and industrial applications. Sintered clutch discs are predominantly used for heavy-duty trucks, vessels, tractors and other agricultural machines. Research facilities such as universities and institutes take advantage of the short sinter cycles, which speeds the research process.

Mounting process is carried on LABOPRESS-3 machine(fig 3.3.2c) and place the cut face of the specimen facing the platform(Fig 3.3.2a). Press the downward button and allow the platform to

reach the bottom. Measure the amount of Bakelite that you need to make the mounting and put it inside the place created by the downward movement of the platform and close the top part of the machine using top closure. Press the upward button on the panel and wait to hear a click sound. Set the force to 20kN, heating time to 6 mins and cooling to 3 mins by using the regulators on the front panel. Press the green button and wait for machine to carry out pressing process and later loosen the top closure. Press the upward button and place the Bakelite specimen mounting is ready.(Fig 3.3.2b)

Fig 3.3.2a Before Hot moulding process

Fig 3.3.2b After Hot moulded

Fig 3.3.2c LABOPRESS-3, machine used for hot pressing

3.3.3 GRINDING

The use of Premium SiC abrasive paper is the most efficient and practical technique for grinding metallic metallographic specimens. Although many qualities of silicon carbide are readily available, only the premium grade SiC powder provides the most consistent results and highest grinding rates.[3]

This crystal structure makes the premium silicon carbide powder an ideal abrasive for cutting and grinding because of its high hardness and sharp edges. The resulting Premium SiC abrasive is an excellent abrasive for maximizing cutting rates while minimizing surface and subsurface damage. For metallographic preparation, Premium SiC abrasives are applied or coated onto abrasive grinding papers. To increase the durability as well as to improve the ability to remove used papers, a thin mylar film backing is bonded to the Premium SiC paper. [3]

Premium SiC abrasive paper is available in all common grit sizes ranging from 60 grit to 1200 grit.[3]

It's very important to have a very good surface finished surface inorder to see the grain boundaries and in here I have used P320, P400, P600, P800, P1200, P2500, P4000(Table 3.3.3)(Fig 3.3.3b) and LABOPOL-5 machine. (Fig 3.3.3a)

Fig 3.3.3a LABOPOL-5, machine on which grinding process is carried

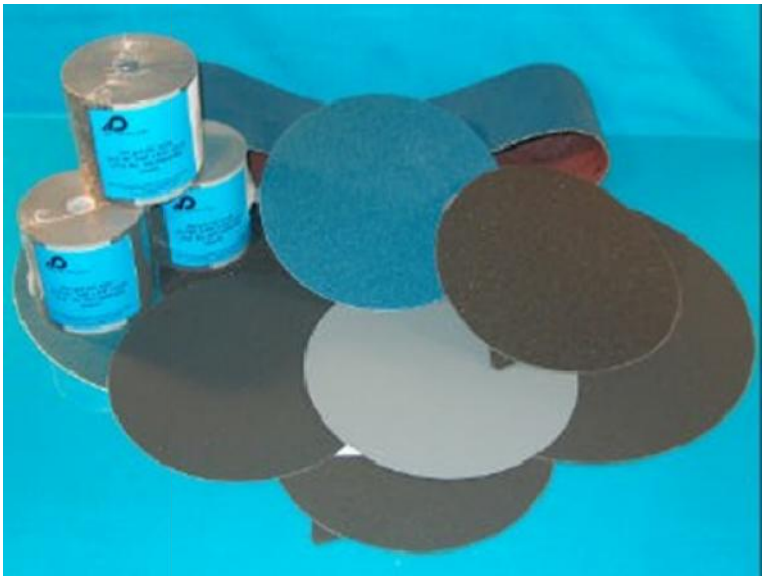


Fig 3.3.3b Grinding papers

Table 3.3.3 Different types of Grinding papers

Standard ANSI grit	European (P-Grade)	Median Diameter (microns)	Surface Roughness on Steel, Rc 30
60	P60	250	-
80	P80	180	1140
120	P120	106	1050
180	P180	75	880
240	P220	63	300
320	P360	40.5	230
400	P800	25.8	120
600	P1200	15.3	110
800	P2400	6.5	25
1200	P4000	2.5	20

3.3.4 POLISHING

Polishing is the process of creating a smooth and shiny surface by rubbing it or using a chemical action, leaving a surface with a significant specular reflection. In some materials (such as metals, glasses, black or transparent stones) polishing is also able to reduce diffuse reflection to minimal values. When an unpolished surface is magnified thousands of times, it usually looks like mountains and valleys. By repeated abrasion, those "mountains" are worn down until they are flat or just small "hills." The process of polishing with abrasives starts with coarse ones and graduates to fine ones.[2]

The strength of polished products is normally higher than their rougher counterpart owing to the removal of stress concentration present in the rough surface. They take the form of corners and other defects which magnify the local stress beyond the inherent strength of the material. Polishing with very fine abrasive differs physically from coarser abrasion, in that material is removed on a molecular level, so that the rate is correlated to the boiling point rather than to the melting point of the material being polished.

We tend to polish the specimen as the surface obtained from the grinding process still possess some scratches and we must eliminate them. In here, I carried polishing with 9µ, 6µ, 1µ Diamond paste (Fig 3.3.4a),(Fig 3.3.4b) on LABOPOL-5 machine.(Fig 3.3.4c)

Fig 3.3.4a Different diamond pastes(6µ, 1µ, 9µ)

Fig 3.3.4b The cloth that's used on metal plates whilst polishing

Fig 3.3.4c LABOPOL-5, machine on which polishing process is carried

3.3.5 ETCHING

Etching is traditionally the process of using strong acid or mordant to cut into the unprotected parts of a metal surface to create a design in intaglio in the metal. [6]Etching by goldsmith and other metal-workers in order to decorate metal items such as guns, armor, cups and plates has been known in Europe since the Middle age at least, and may go back to antiquity. The elaborate decoration of armor, in Germany at least, was an art probably imported from Italy around the end of the 15th century—little earlier than the birth of etching as a printmaking technique. The switch to copper plates was probably made in Italy, and thereafter etching soon came to challenge engraving as the most popular medium for artists in printmaking. Its great advantage was that, unlike engraving where the difficult technique for using the burin requires special skill in metalworking, the basic technique for creating the image on the plate in etching is relatively easy to learn for an artist trained in drawing. On the other hand, the handling of the ground and acid need skill and experience, and are not without health and safety risks, as well as the risk of a ruined plate. Prior to 1100 AD, the New World Hohokam independently utilized the technique of acid etching in marine shell designs.[7]

This process is important as we oxidize the grain boundaries with an etchant so that it's seen properly and here we are using KALLINGS as our etchant (Fig 3.3.5a). Clean the specimen with ethanol or acetone, so that you get rid of all dust particles and heat the kallings upto 45° C (Hot etchant).Carry the etching process for about 30 minutes. Note: We must be periodically checking the specimen under microscope because if we overdo it then we are likely to burn the surface of the specimen. (Fig 3.3.5b)(Fig 3.3.5c)

Fig 3.3.5a Kalling's solution

CHEMICAL REPRESENTATION OF KALLINGS

5gm of Copperchloride+100ml of ethanol+100ml of HCl=KALLING's

Fig 3.3.5b Before etching

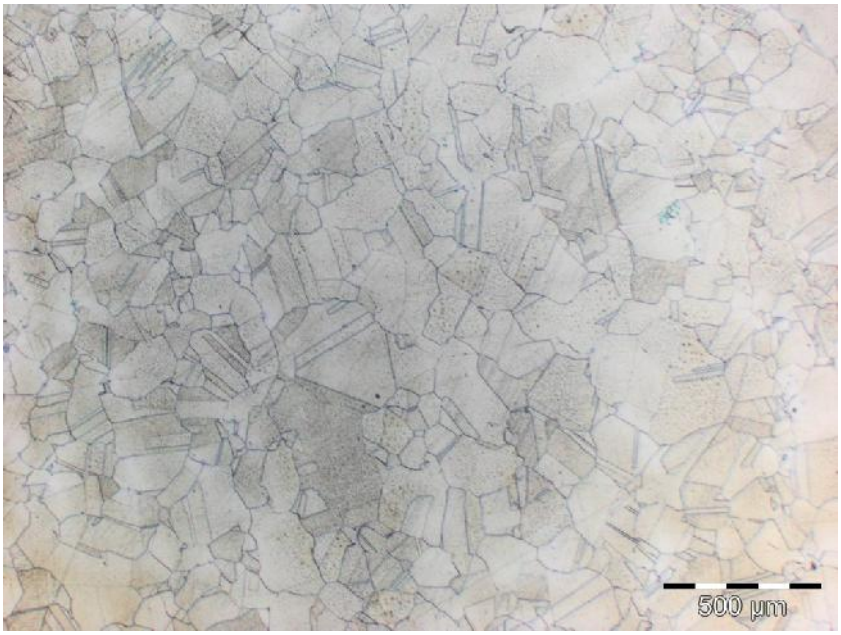


Fig 3.3.5c After Etching using an etchant, the grain boundries are corroded and appear as in the figure shown above

3.4 METALLOGRAPHY (OM & SEM)

Metallography is the study of the physical structure and components of metals, typically using microscopy. The surface of a metallographic specimen is prepared by various methods of grinding, polishing, and etching. After preparation, it is often analyzed using optical or electron microscopy. Using only metallographic techniques, a skilled technician can identify alloys and predict material properties. Prepared specimens should be examined with the unaided eye after etching to detect any visible areas that have responded to the etchant differently from the norm as a guide to where microscopical examination should be employed. Light optical microscopy (LOM) examination should always be performed prior to any electron metallographic (EM) technique, as these are more time-consuming to perform and the instruments are much more expensive.[8]

The optical microscope,(Fig 3.4a) often referred to as light microscope, is a type of microscope which uses visible light and a system of lenses to magnify images of small samples. Optical microscopes are the oldest design of microscope and were possibly invented in their present compound form in the 17th century. Basic optical microscopes can be very simple, although there are many complex designs which aim to improve resolution and sample contrast. The image from an optical microscope can be captured by normal light-sensitive cameras to generate a micrograph. Originally images were captured by photographic film but modern developments in CMOS and charge-coupled device (CCD) cameras allow the capture of digital images. Purely digital microscopes are now available which use a CCD camera to examine a sample, showing the resulting image directly on a computer screen without the need for eyepieces.[8]

A scanning electron microscope (SEM)(Fig 3.4b) is a type of electron microscope that produces images of a sample by scanning the surface with a focused beam of electrons. The electrons interact with atoms in the sample, producing various signals that contain information about the sample's surface topography and composition. The electron beam is scanned in a raster scan pattern, and the beam's position is combined with the detected signal to produce an image. SEM can achieve resolution better than 1 nanometer. Specimens can be observed in high vacuum in conventional SEM, or in low vacuum or wet conditions in variable pressure or environmental SEM, and at a wide range of cryogenic or elevated temperatures with specialized instruments. The most common SEM mode is detection of secondary electrons emitted by atoms excited by the electron beam. The number of secondary electrons that can be detected depends, among other things, on

specimen topography. By scanning the sample and collecting the secondary electrons that are emitted using a special detector, an image displaying the topography of the surface is created.[9]

Note: Make sure that the specimen is cleaned with ethanol or acetone and dry it properly before you take the specimen for metallograpy.

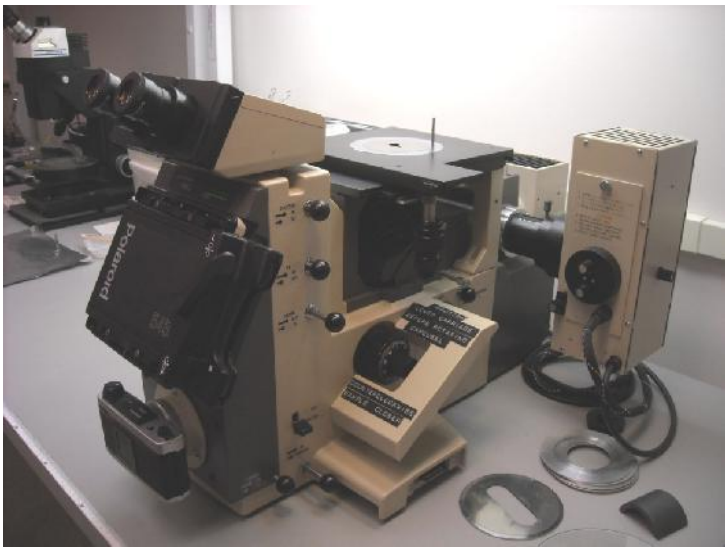


Fig 3.4.a A picture of OM[20]

Fig3.4.b A picture of SEM[21]

3.5 SEMI QUANTITATIVE ANALYSIS

Energy-dispersive X-ray spectroscopy (EDS, EDX, EDXS or XEDS), sometimes called energy dispersive X-ray analysis (EDXA) or energy dispersive X-ray microanalysis (EDXMA), is an analytical technique used for the elemental analysis or chemical characterization of a sample. It relies on an interaction of some source of X-ray excitation and a sample. Its characterization capabilities are due in large part to the fundamental principle that each element has a unique atomic structure allowing a unique set of peaks on its electromagnetic emission spectrum.[11]. In some instances the conditions for reliable quantitative data are not met, but qualitative data is not enough to answer the questions at hand. In these situations, there is a third option. Semi-quantitative data processing allows the user to compare spectral data from samples in order to obtain information regarding the relative concentrations of elements from sample to sample. While this method does not provide absolute concentration values, it can be used to ascertain relative element concentrations between samples; for example, this method would provide information such as "sample A contains approximately 20% more molybdenum than sample B." This sort of data is extracted by calculating the area under each peak of interest, which is equivalent to the number of counts. This type of data is appropriate in situations where a calibration and/or samples of known concentrations do not exist, but comparing the samples in terms of element concentration is necessary.[23](fig 3.5)

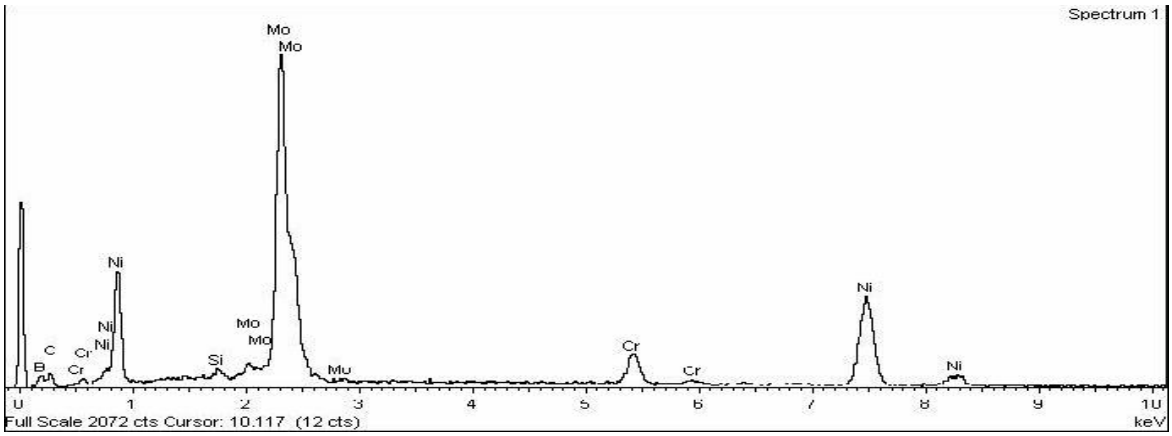


Fig 3.5 Picture of semi quantitative analysis of Haynes-242(As received)

3.6 GRAIN SIZE MEASUREMENT

Metals, except in a few instances, are crystalline in nature and, except for single crystals; they contain internal boundaries known as grain boundaries. When a new grain is nucleated during processing (as in solidification or annealing after cold working), the atoms within each growing grain are lined up in a specific pattern that depends upon the crystal structure of the metal or alloy. With growth, each grain will eventually impinge on others and form an interface where the atomic orientations are different. As early as the year 1900, it was well known that most mechanical properties were improved as the size of the grains decreased. A few notable exceptions exist where a coarse grain structure is desired. Alloy composition and processing must be controlled to achieve the desired grain size. Metallographers examine polished cross sections of specimens from appropriate locations to determine the grain size.

Intercept method. Its one of the ways in which grain size can be determined. In this method several vertical and horizontal lines are drawn on the OM image of the specimen and no of times the lines intersect the grain boundaries are noted and the following formulas are used to determine avg grain size. Compare the grain size with ASTM grain number and note down. (Fig 3.6)

$$\text{Lines per intersection (LPR)} = (\text{length of the line in mm}) * \frac{\text{N o li}}{\text{n o t i}}$$

$$\text{LPR} * \frac{\text{v o tl s b}}{\text{le o tl s b i m}}$$

$$\text{Average grain size} = \left\{ \frac{H \quad g \quad s \quad + V \quad G \quad s}{2} \right]$$

Relación de tamaños de grano ASTM E-112		
Promedio diámetro D		
Tamaño de grano g	mm	µm
00	0.508	508
0	0.3592	359.2
0.5	0.3021	302.1
1	0.254	254
1.5	0.2136	213.6
2	0.1796	179.6
2.5	0.151	151
3	0.127	127
3.5	0.1068	106.8
4	0.0898	89.8
4.5	0.0755	75.5
5	0.0635	63.5
5.5	0.0534	53.4
6	0.0449	44.9
6.5	0.0378	37.8
7	0.0318	31.8
7.5	0.0267	26.7
8	0.0225	22.5
8.5	0.0198	19.8
9	0.0159	15.9
9.5	0.0133	13.3
10	0.0112	11.2
10.5	0.0094	9.4
11	0.0079	7.9
11.5	0.0067	6.7
12	0.0056	5.6
12.5	0.0047	4.7
13	0.004	4
13.5	0.0033	3.3
14	0.0028	2.8

Fig 3.6 Relation between average grain size and ASTM grain number[25]

3.7 VICKERS HARDNESS NUMBER

The Vickers test is often easier to use than other hardness tests since the required calculations are independent of the size of the indenter, and the indenter can be used for all materials irrespective of hardness (Fig3.7). The basic principle, as with all common measures of hardness, is to observe the questioned material's ability to resist plastic deformation from a standard source. The Vickers test can be used for all metals and has one of the widest scales among hardness tests. The unit of hardness given by the test is known as the Vickers Pyramid Number (HV) or Diamond Pyramid Hardness (DPH). The hardness number can be converted into units of Pascals, but should not be confused with pressure, which also has units of Pascals. The hardness number is determined by the load over the surface area of the indentation and not the area normal to the force, and is therefore not pressure. [10]

Fig 3.7.The pyramidal diamond indenter [24]

4. RESULTS

4.1 INITIAL MICROSTRUCTURAL CHARACTERISATION

Grain size plays an important role in determining the mechanical properties of the sample as it affects yield stress, machinablity, hardeness and etc. The result obtained under 3 different condition i.e as received, solutionised and aged are given below.

4.1.1 AS RECEIVED

Grain size number of solutionised specimen was found out to be 3(ASTM) by using intercept method. Fig 4.1.1 represents the microstructure of sample prepared at room temperature and not many precipitates are seen and grain size is smaller.

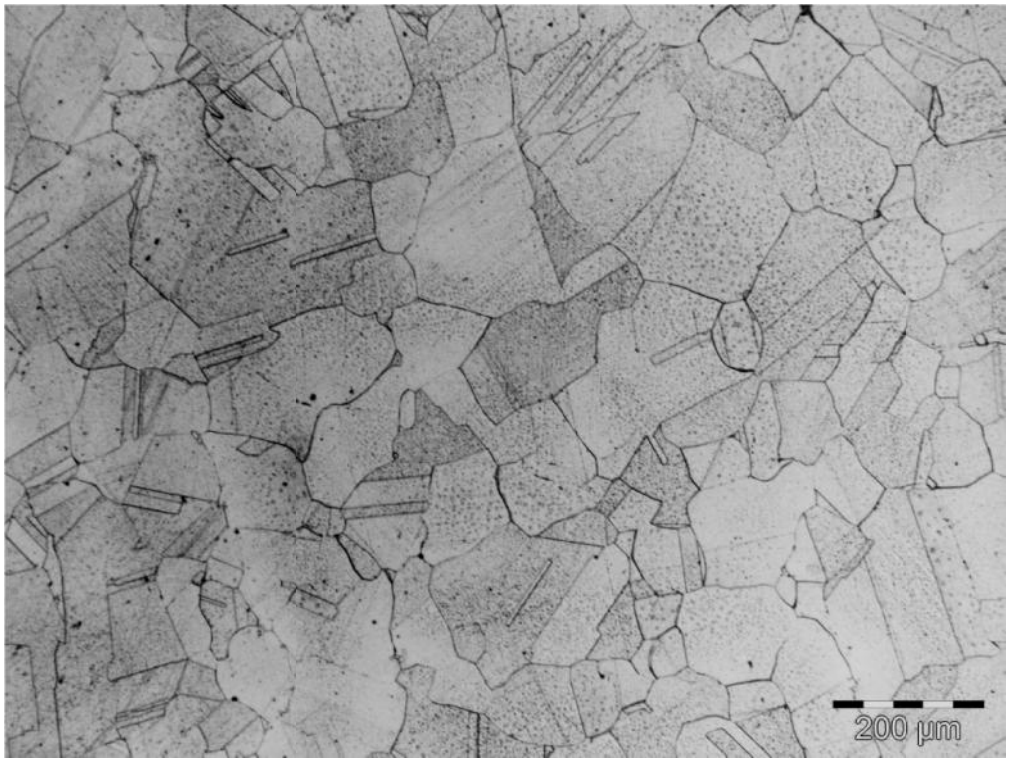


Fig 4.1.1 Microstructure of an as received sample

4.1.2 SOLUTIONISED

Grain size number of solutionised specimen was found out to be 3(ASTM) by using intercept method. Fig 4.1.2 represents the microstructure of sample prepared at 1066˚C for 30 minutes. The grains look very similar to as received ones(see fig 4.1.1) and smaller the aged ones(see fig 4.1.3)

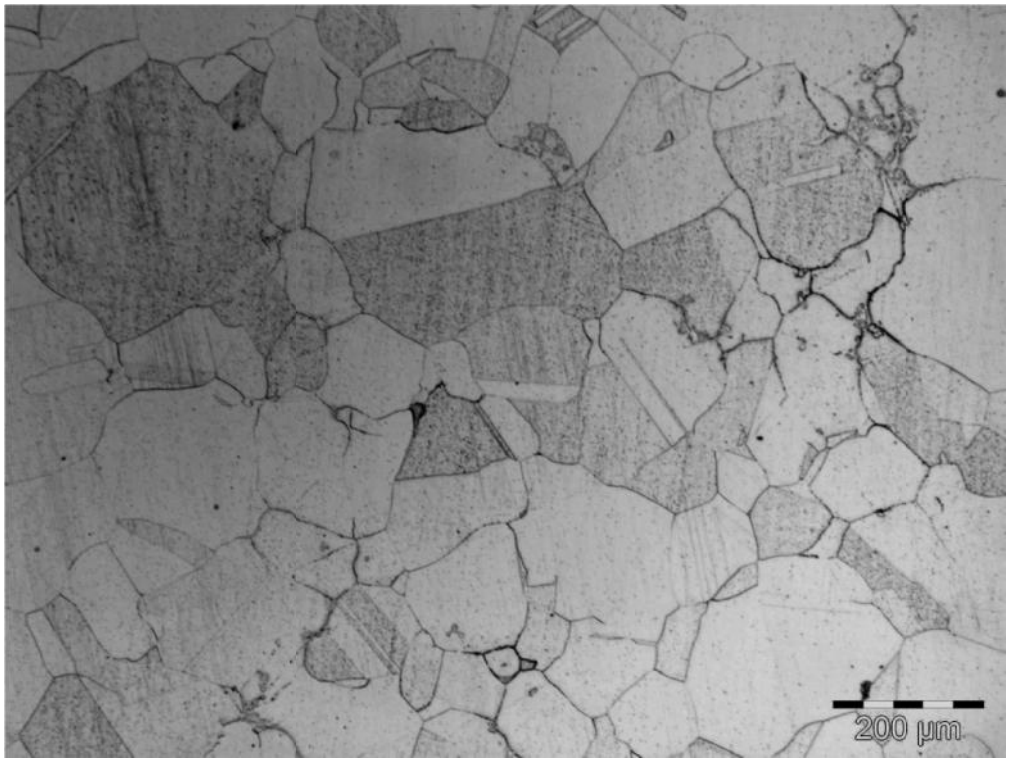


Fig 4.1.2 Microstructure of solutionised sample

4.1.3 AGED

Grain Size number of aged sample was found out to be 2.5 using intercept method. Fig 4.1.3 represents the microstructure of sample prepared at 640˚C for 24H.The grains looks comparatively bigger than as received and solutionised sample (see figure 4.1.1,4.1.2).

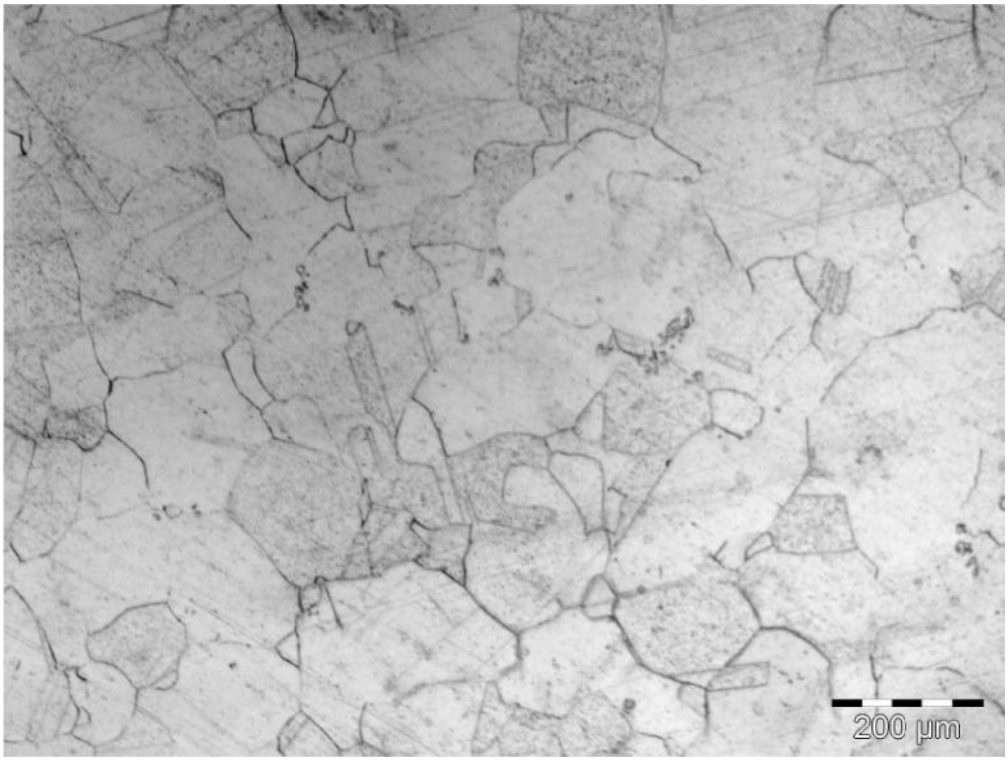


Fig 4.1.3 Microstructre of Aged sample

4.1.4 VICKERS HARDNESS

Table 4.1.4 represents the HV of As received solutionised and aged samples and from the table it can be inferred that As received and solutionised sample have similar HV. General trend is observed in the case of aging as a lot of precipitates are present.

Table4.1.4 Represents hardness measurements of different samples (as received, solutionised, aged)

Kg	Type of Specimen	HV(average values)
0.3	As Received	226.2±10
0.3	Solutionised	235.5±11
0.3	Aged	622.6±16

4.1.5 GRAPHS

Fig 4.1.5a represents histogram between three different specimens i.e as received, solutionised and aged with their respective grain size number. As seen from the histogram it's known to us that as received and solutionised samples have same grain size number. There is an increase in grain size of an aged sample as it's given ample amount of time inside the furnace during the aged thermal treatment for grain growth and hence there is decline in grain size number.

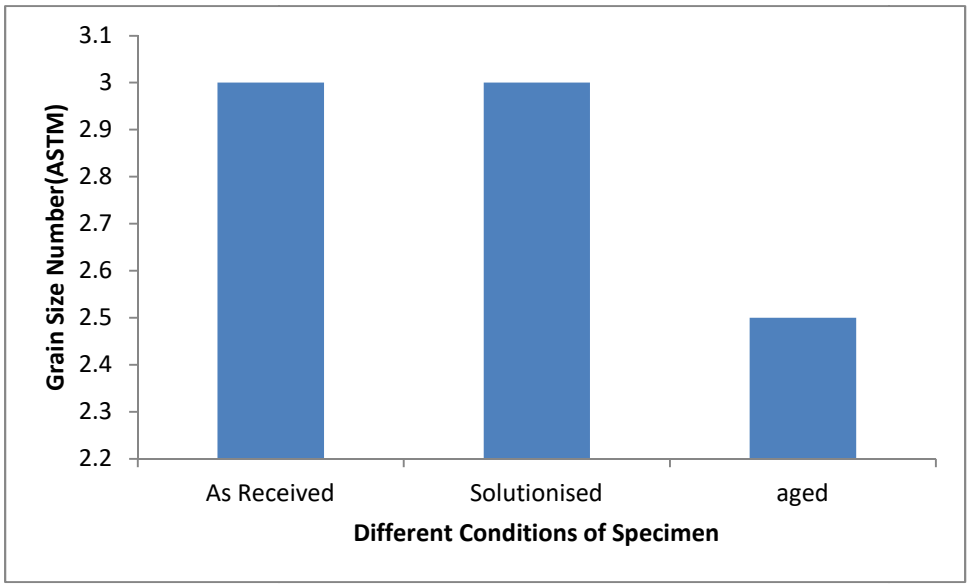


Fig 4.1.5a Graph plotted between type of specimen Vs ASTM grain size number.

Fig 4.1.5b represents histogram between three different specimens i.e as received, solutionised and aged with their respective Vickers hardness number. It can be seen that as received and solutionised samples have similar Vickers hardness number. There is an increase in HV for an aged sample as it has a lot of precipitates and these precipitates amplifies the hardness of the material.

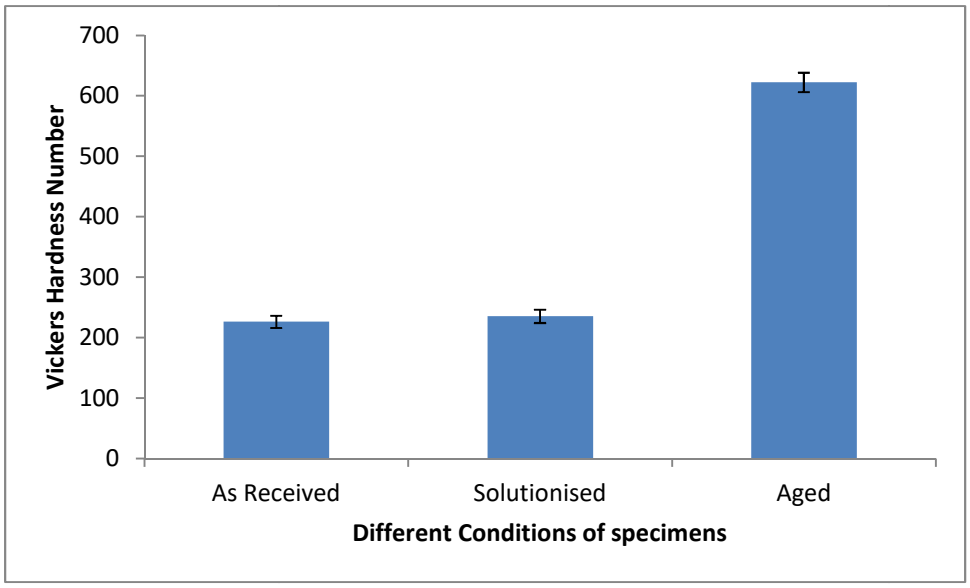


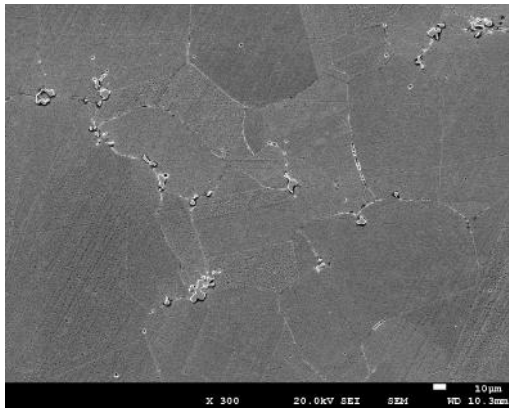
Fig 4.1.5b plot that shows variation of HV with respect

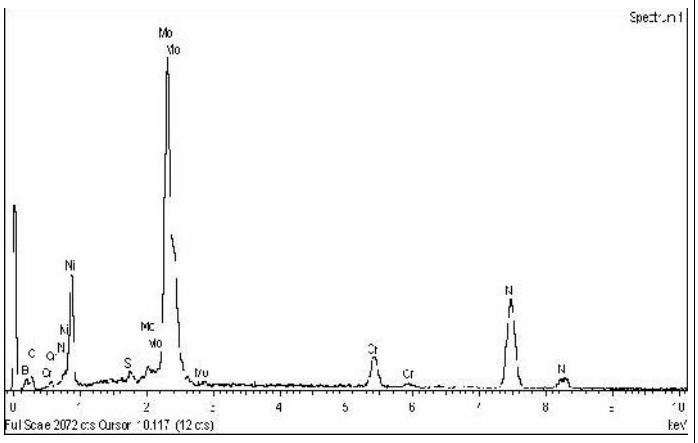
to different specimens

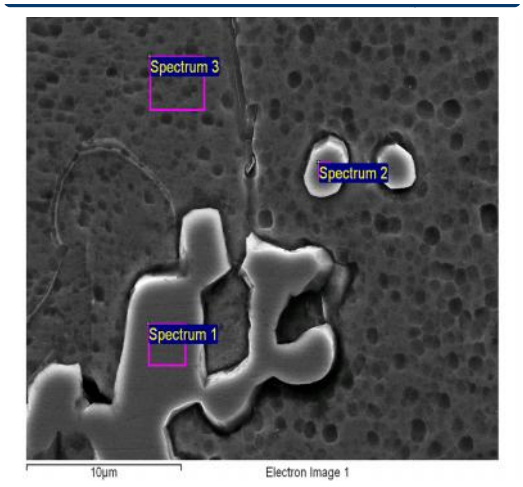
4.2 SEMI QUANTITATIVE ANALYSIS

4.2.1 AS RECEIVED

Fig 4.2.1 represents specimen in as received condition and its clearly visible that not many precipitates that are present, very similar to that of solutionised specimen(see figure 4.2.2a)







Processing option : All elements analysed (Normalised)

Spectrum	In stats.	B	C	Si	Cr	Fe	Ni	Mo	Total
Spectrum 1	Yes	10.08	10.49	0.50	4.87		27.76	46.30	100.00
Spectrum 2	Yes	14.23	8.92		4.50	0.67	27.16	44.53	100.00
Spectrum 3	Yes		9.35		7.38	0.89	55.18	27.21	100.00
Max.		14.23	10.49	0.50	7.38	0.89	55.18	46.30	
Min.		10.08	8.92	0.50	4.50	0.67	27.16	27.21	

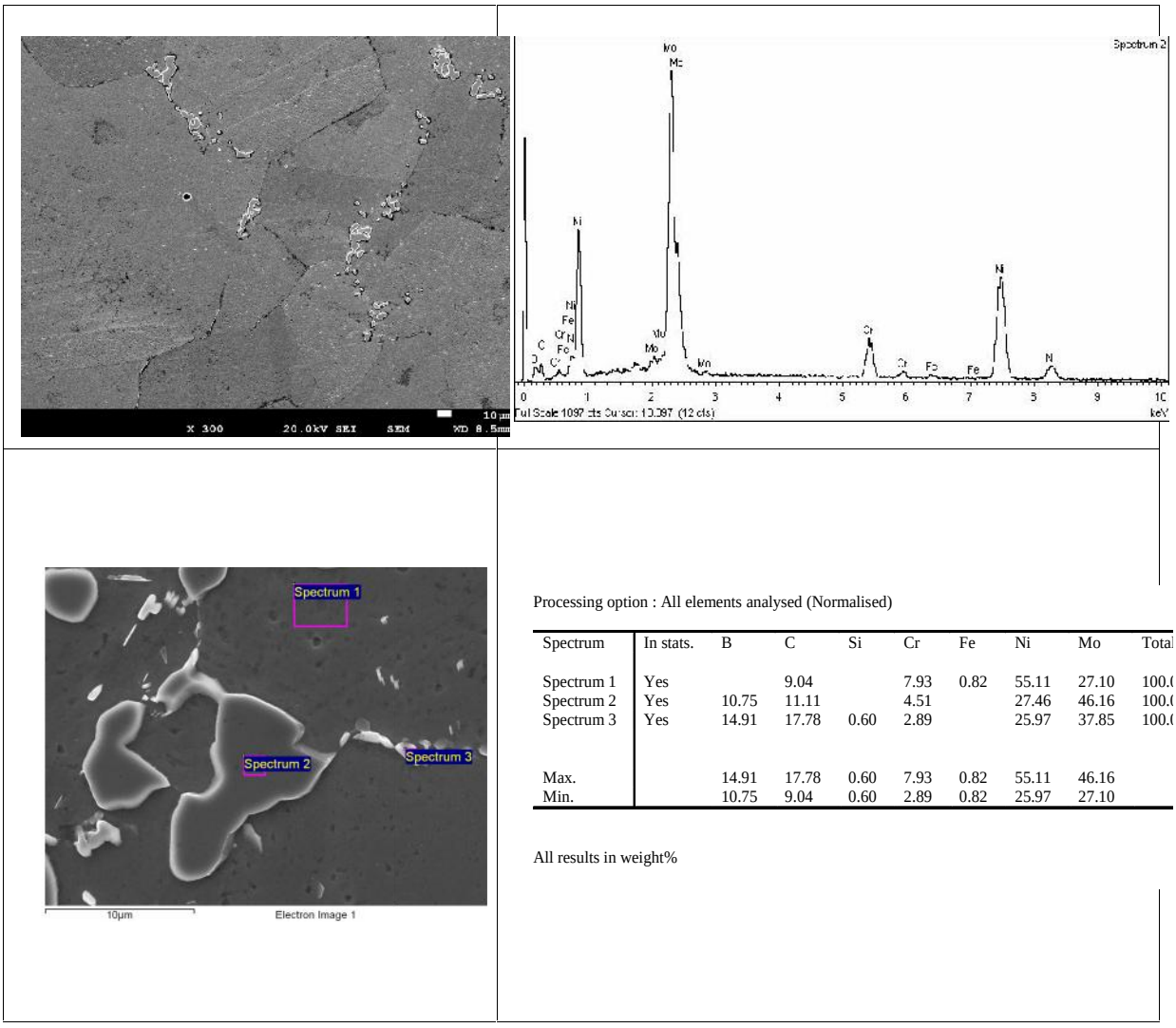
All results in weight%

Fig 4.2.1 (clockwise)SEM image at 300X of as received,

EBSD analysis at 3 different points

4.2.2 SOLUTIONISED

Fig 4.2.2 represents solutionised specimen and it's clearly observed that there are less precipitates that are present as this process is carried at higher temperature; most of the precipitates gets dissolved.



Processing option : All elements analysed (Normalised)

Spectrum	In stats.	B	C	Si	Cr	Fe	Ni	Mo	Total
Spectrum 1	Yes		9.04		7.93	0.82	55.11	27.10	100.(
Spectrum 2	Yes	10.75	11.11		4.51		27.46	46.16	100.(
Spectrum 3	Yes	14.91	17.78	0.60	2.89		25.97	37.85	100.(
Max.		14.91	17.78	0.60	7.93	0.82	55.11	46.16	
Min.		10.75	9.04	0.60	2.89	0.82	25.97	27.10	

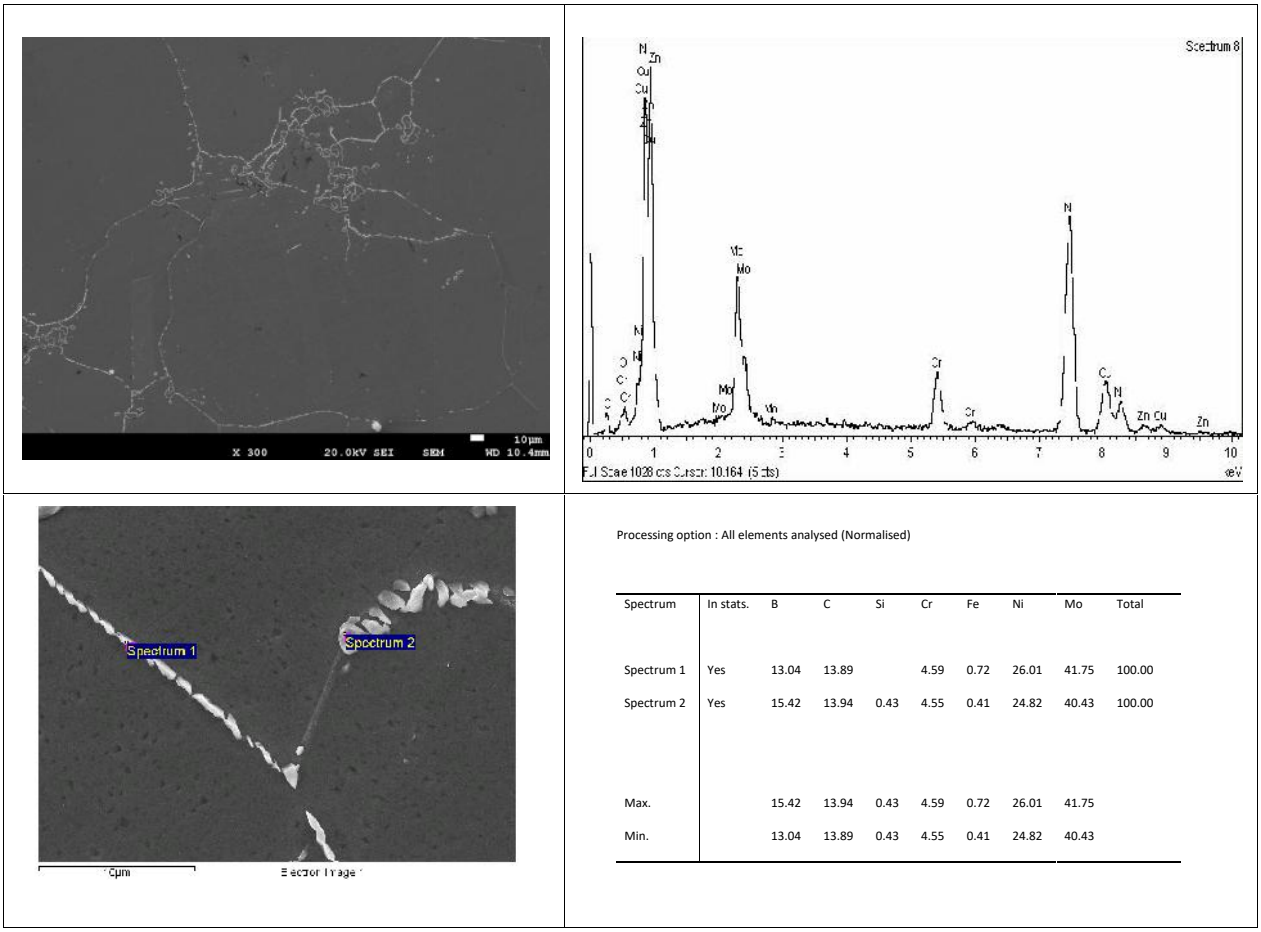
All results in weight%

Fig 4.2.2 (clockwise)SEM image at 300X of solutionised,

EBSD analysis at 3 different points

4.2.3 AGED

Fig 4.2.3 represents EDX on aged sample; it's observed that comparatively alot more precipates are seen along the grain boundries and as there are a lot of precipitates seen and most of precipitates are carbides hence HV is bound to increase(see table 4.1.5)



Spectrum	In stats.	B	C	Si	Cr	Fe	Ni	Mo	Total
Spectrum 1	Yes	13.04	13.89		4.59	0.72	26.01	41.75	100.00
Spectrum 2	Yes	15.42	13.94	0.43	4.55	0.41	24.82	40.43	100.00
Max.		15.42	13.94	0.43	4.59	0.72	26.01	41.75	
Min.		13.04	13.89	0.43	4.55	0.41	24.82	40.43	

Fig 4.2.3 (clockwise)SEM image at 300X of aged and

EBSD analysis at 2 different points

4.3 HOT COMPRESSED TESTS

4.3.1 HOT FLOW CURVES

The hot flow curves were determined by hot compression tests and the following figures represents the hot flow curves at different temperature and strain rates.As observed from these following graphs, the peak stress is decreasing with increase in temperaturas.By comparing the different plots, the effect of the strain rate can be analysed for a given temperature and behaviour is as expected : higher strain rates are associated to higher stresses.

Fig 4.3a shows behavior of the hot deformed samples at different temperature keeping strain rate constant ($0.001s^{-1}$). Here its observed that there is no significant dip for lower temperatures like 900˚C -950˚C whereas there is considerable dip at higher temperatures and might be due to the necklace recrystallization observed at 1050˚C (see fig 4.2.2.7)

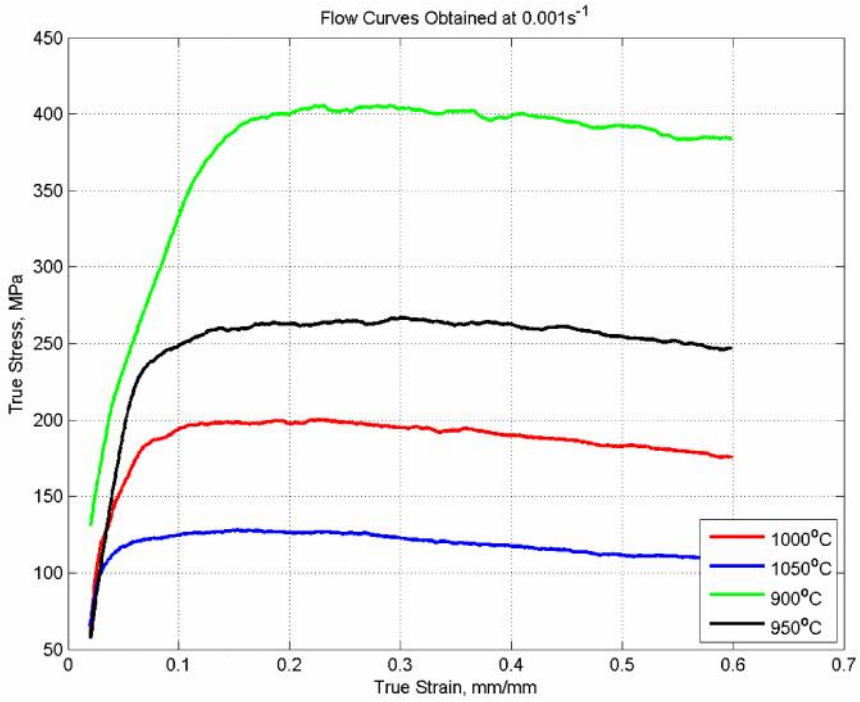


Fig 4.3a Flow curve of $0.001s^{-1}$ with different temperature. This curve is plotted between true stress and true strain of hot compressed specimens at different temperatures(900˚C - 1050˚C) at constant strain rate.

Fig 4.3b shows behavior of the hot deformed samples at different temperature keeping strain rate constant ($0.01s^{-1}$).As observed in Fig 4.3a similar behavior is observed in here. There is considerable decrease in slope but not to greater extent which could mean that no full recrystallization but only necklace recrystallization is taking place.

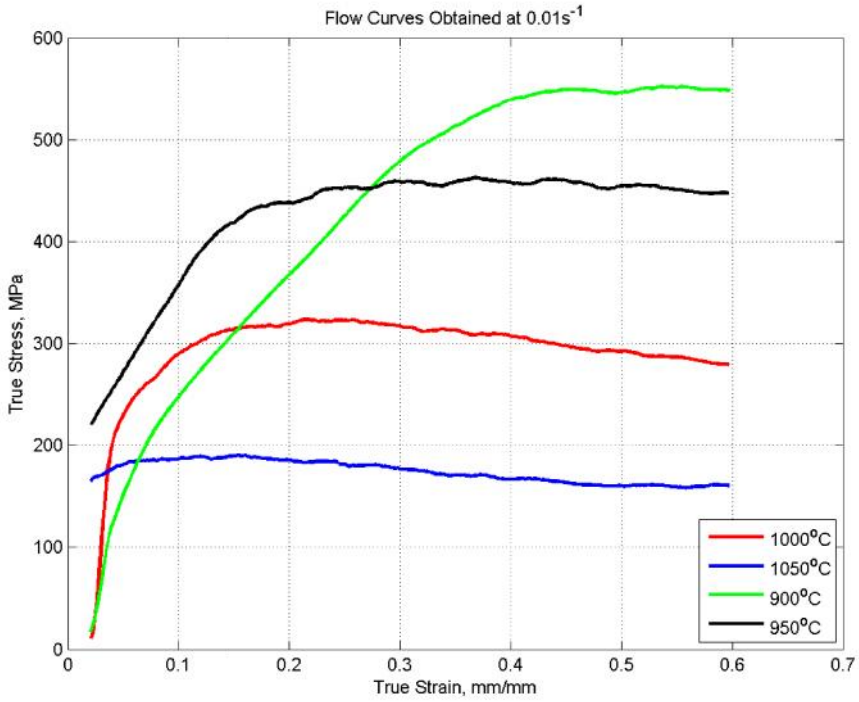


Fig 4.3b Flow curve of $0.01s^{-1}$ with different temperature.This curve is plotted between true stress and true strain of hot compressed specimens at different temperatures(900˚C - 1050˚C) at constant strain rate.

Fig 4.3c shows behavior of the hot deformed samples at different temperature keeping strain rate constant ($0.05s^{-1}$). There is no significant dip in any curves observed hence could say that there is only recovery taking place at this strain rate and its further bolstered by the microscopic images (see fig 4.2.2.2, 4.2.2.6)

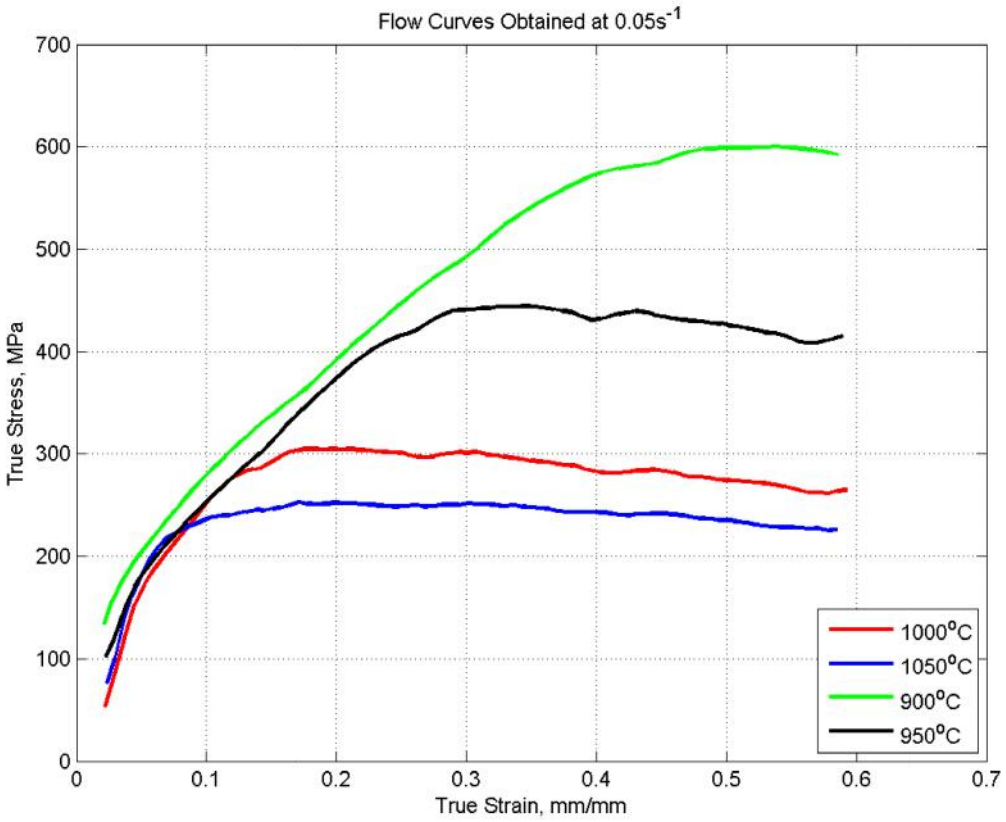


Fig 4.3c Flow curve of $0.05s^{-1}$ with different temperature. This curve is plotted between true stress and true strain of hot compressed specimens at different temperatures (900˚C - 1050˚C) at constant strain rate.

Fig 4.3d shows behavior of the hot deformed samples at different temperature keeping strain rate constant($0.1s^{-1}$) .Based on previous graphs (see fig 4.3a,4.3b,4.3c) it can be inferred that with increase in strain rate, the peak stress value would also increase siginificantly and there is only recovery taking place.

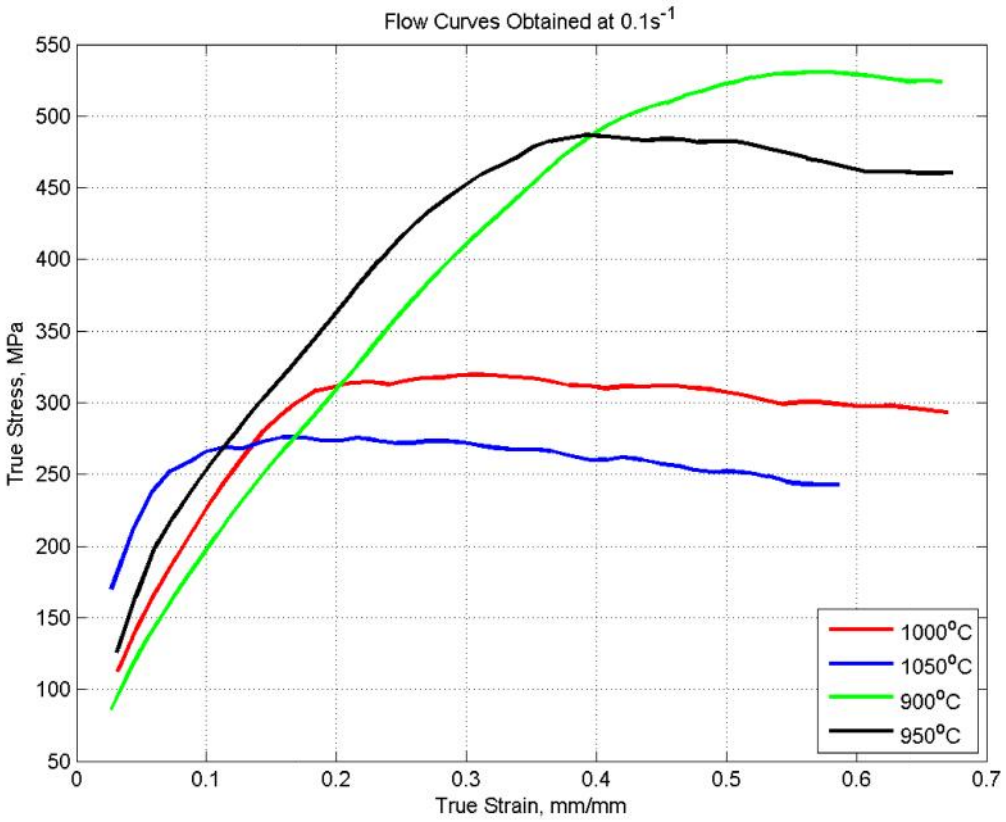


Fig 4.3d Flow curve of $0.1s^{-1}$ with different temperature. This curve is plotted between true stress and true strain of hot compressed specimens at different temperatures (900˚C - 1050˚C) at constant strain rate.

Fig 4.3e shows behavior of the hot deformed samples at different temperature with varying strain rate.Its clearly seen that peak stress is increasing with increase in strain rate and lower the temperature higher the peaker stress as we need to apply more pressure to deform the sample.

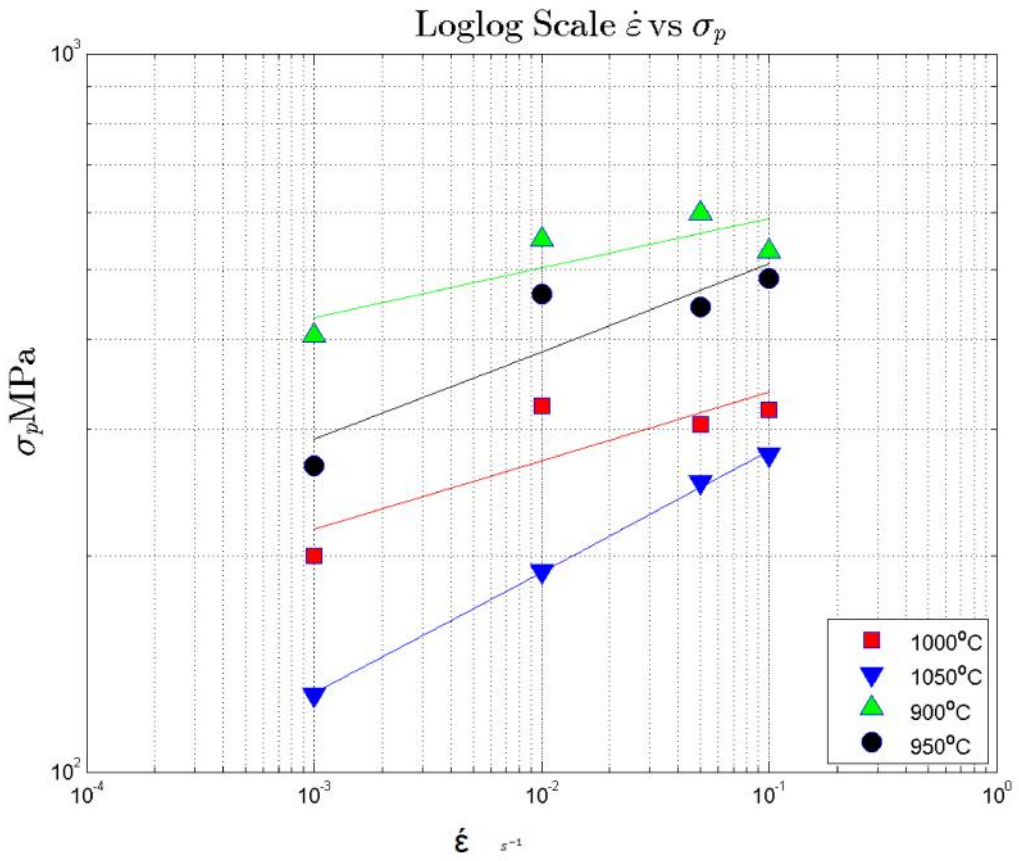


Fig 4.3e Strain rate Vs peak stress.The above graph is plotted in logarithmic scale btwn Strain rate and peak stress obtained during hot compression tests of different specimens at different temperature and strain rate.

4.3.2 EVOLUTION MICROSTRUCTURAL EVOLUTION

4.3.2.1 900˚C at $0.1s^{-1}$

Figure 4.3.2.1 represents the microstructure of the sample tested at 900C at $0.1s^{-1}$.Its observed that grain size is really smaller when compared to other temperatures and a lot of twinning has been spotted.

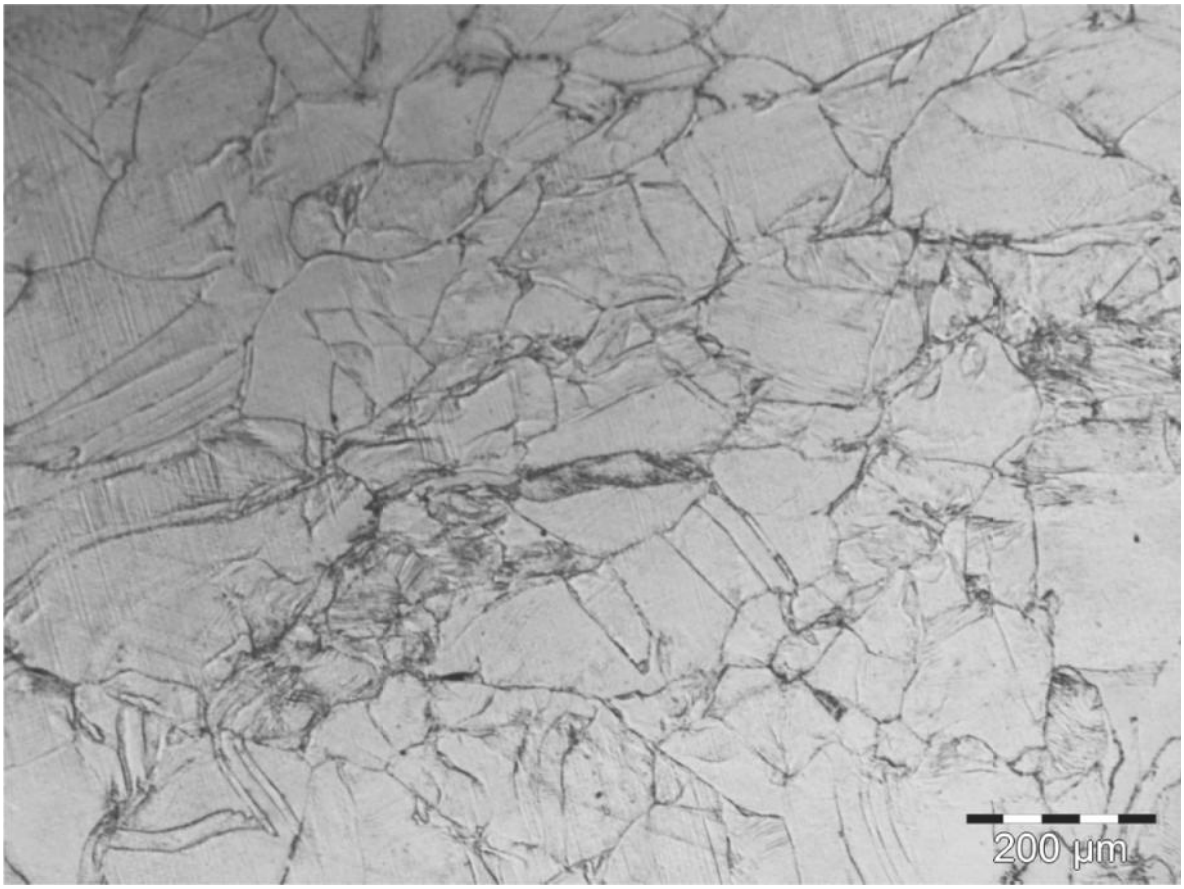


Fig 4.3.2.1 Grain size distribution of hot compressed simple at 900˚C and $0.1s^{-1}$

4.3.2.2 900˚C at $0.05s^{-1}$

Figure 4.3.2.2 represents the microstructure of the sample tested at 900C at $0.05s^{-1}$.Grain size looks comparatively bigger than the ones deformed at same temperature but with higher strain rate.(see figure 4.2.2.1).

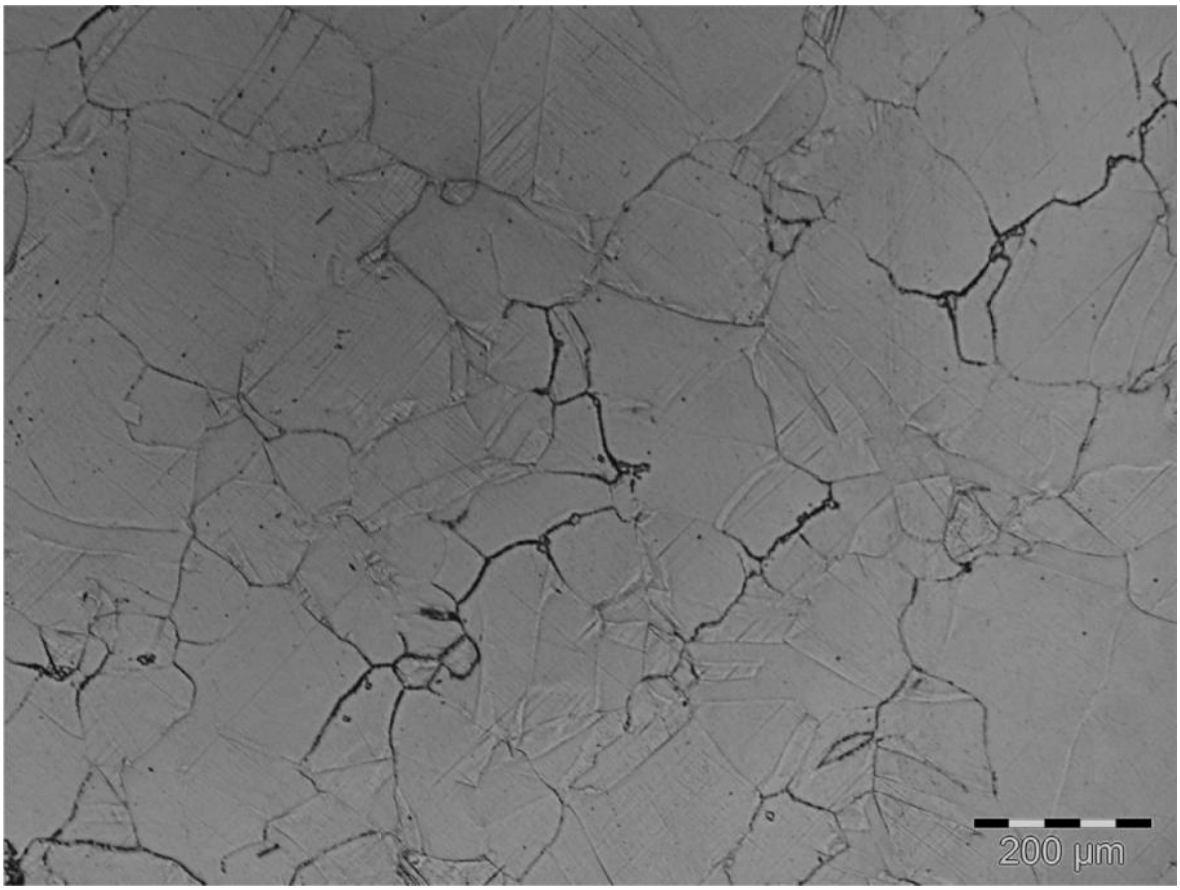


Fig 4.3.2.2 Grain size distribution of hot compressed simple at 900˚C and $0.05s^{-1}$

4.3.2.3 950˚C at $0.01s^{-1}$

Figure 4.3.2.3 represents the microstructure of the sample tested at 950C at $0.01s^{-1}$.Grain size looks similar to the hot deformed samples at lower temperature.

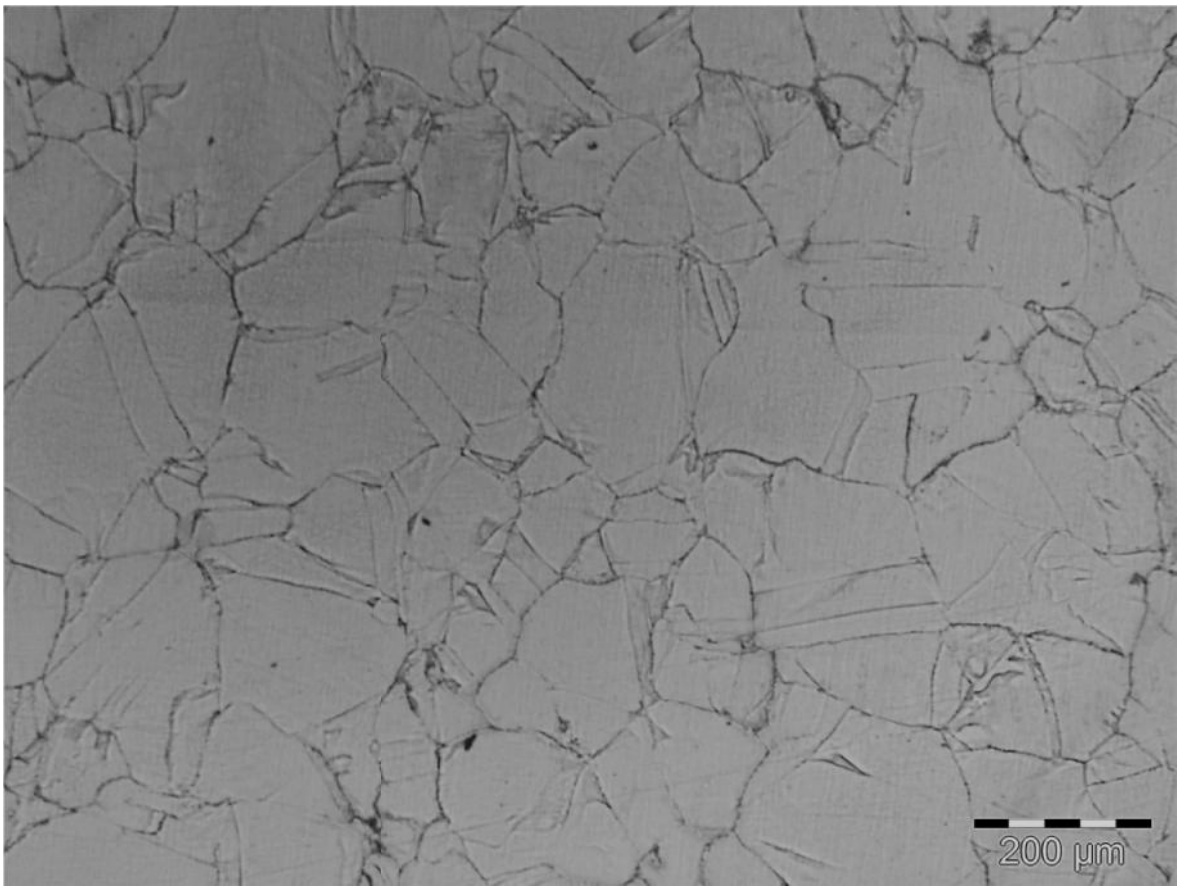


Fig 4.3.2.3 Grain size distribution of hot compressed simple at 950˚C and $0.01s^{-1}$

4.3.2.4 950˚C at $0.1s^{-1}$

Figure 4.3.2.4 represents the microstructure of the sample tested at 950C at 0.1s^{-1}.The grain size is comparatively smaller when compared to hot deformed samples at high temperature.

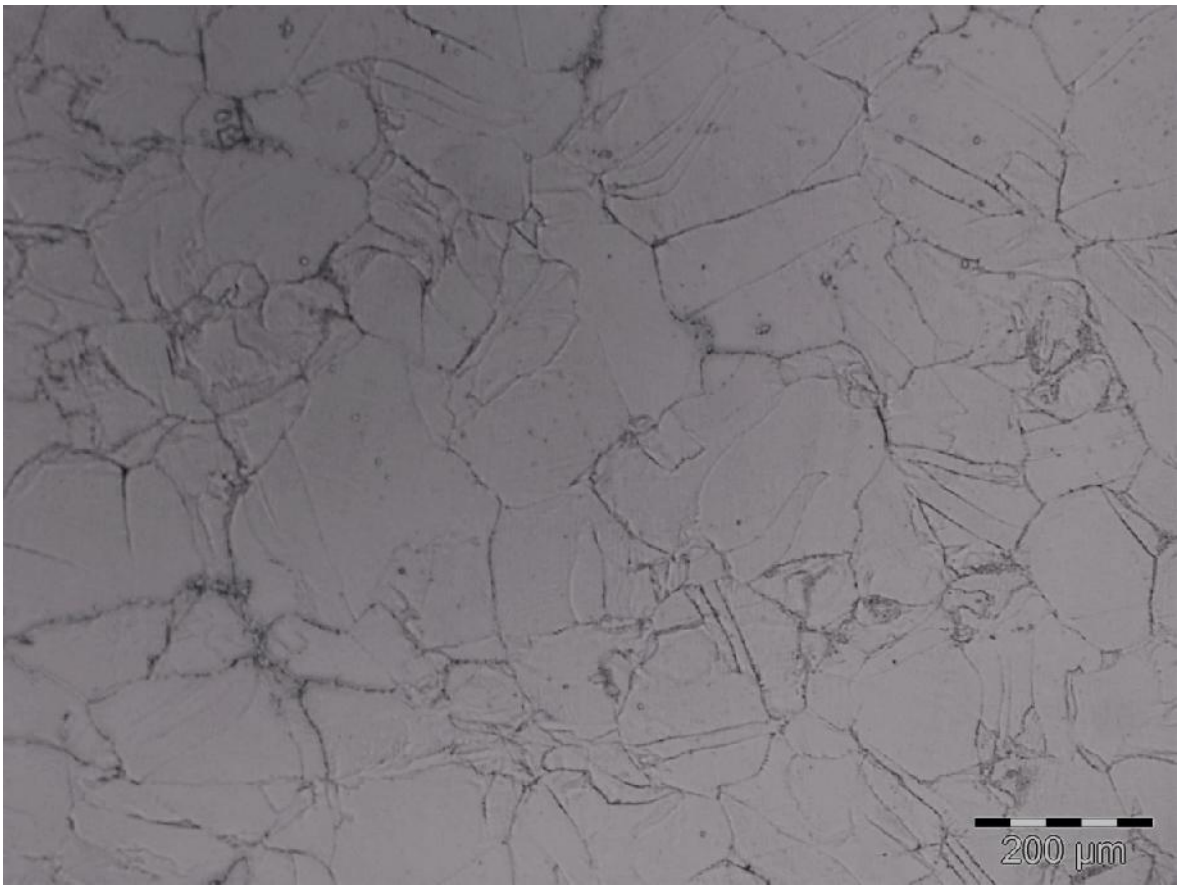


Fig 4.3.2.4 Grain size distribution of hot compressed sample at 950˚C and 0.1s^{-1}

4.3.2.5 1000˚C at $0.01s^{-1}$

Figure 4.3.2.5 represents the microstructure of the sample tested at 1000˚C at $0.01s^{-1}$. The presence of recrystallization by the formation of new grains at grain boundaries, i.e. necklace recrystallization is evident.

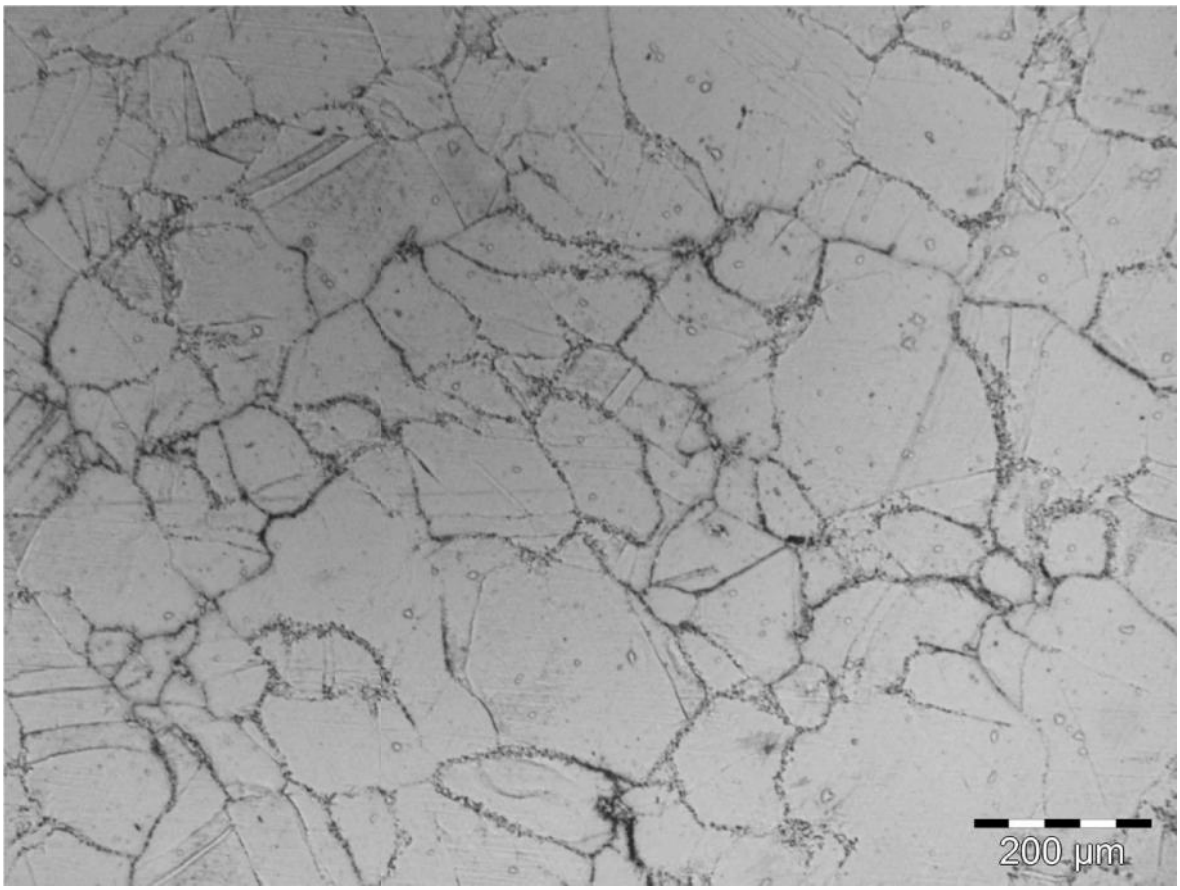


Fig 4.3.2.5 Grain size distribution of hot compressed sample at 1000˚C and $0.01s^{-1}$

4.3.2.6 1000˚C at $0.05s^{-1}$

Figure 4.3.2.6 represents the microstructure of the sample tested at 1000C at $0.05s^{-1}$.Its observed that no necking recrystallization taking place like the one in fig 4.2.2.5 inspite of both being at same temperature, hence strain rate also plays a vital role.

Fig 4.3.2.6 Grain size distribution of hot compressed sample at 1000˚C and $0.05s^{-1}$**.**

4.3.2.7 1050˚C at $0.001s^{-1}$

Figure 4.3.2.7 represents the microstructure of the sample tested at 1050C at $0.001s^{-1}$. The presence of recrystallization by the formation of new grains at grain boundaries, i.e. necklace recrystallization is evident. The size of the new grains is bigger than the ones forming at lower temperatures and/or slower strain rates (see Figure 4.2.2.5).

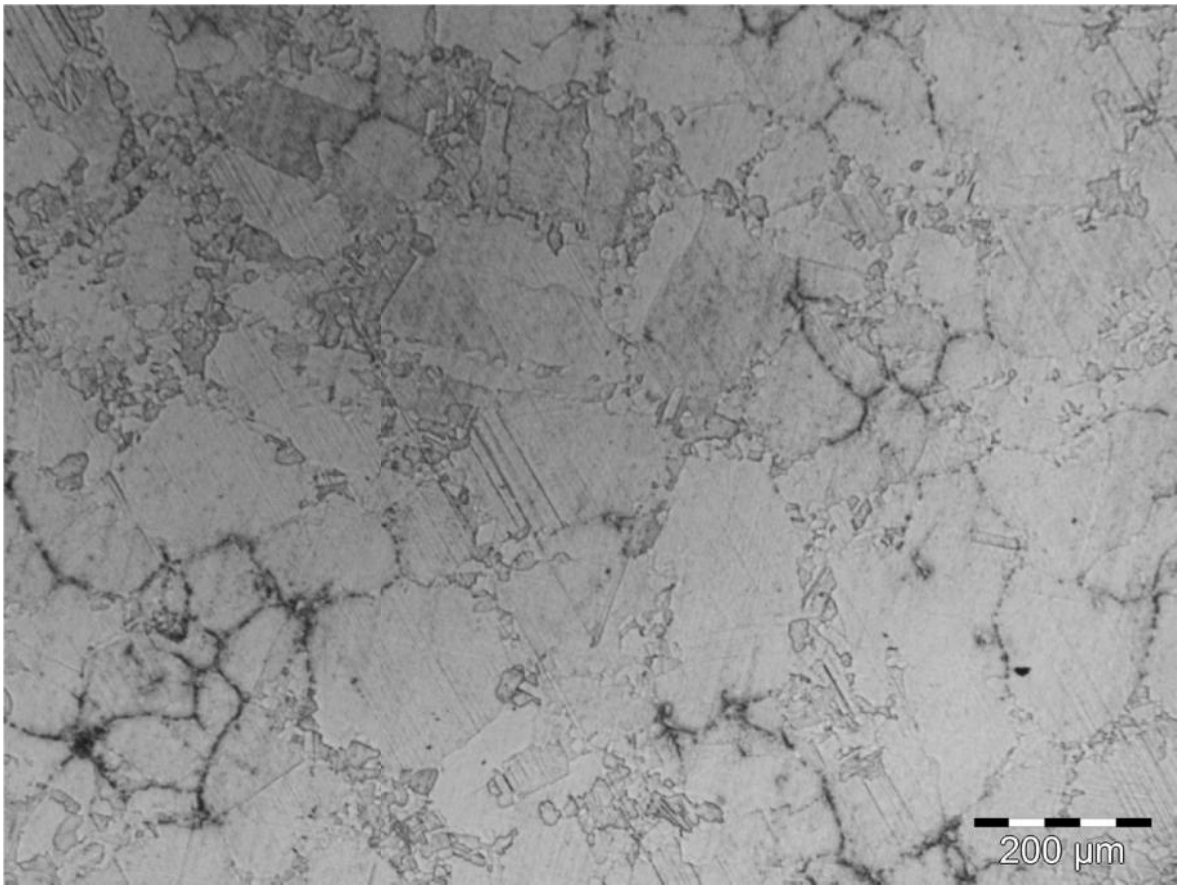


Fig 4.3.2.7 Grain size distribution of hot compressed sample at 1050˚C and $0.001s^{-1}$

4.3.3 VICKERS HARDNESS OF HOT COMPRESSED SPECIMENS

Table 4.3.3 shows the general trend of decrease in hardness as the temperatura increases as the material becomes more and more softned and materials generally deformed at higher strain rates at same temperature tends to have higher hardness.

Table 4.3.3 Table represents HV of different Hot compressed specimens

Kg	Temperature	Strain Rate	HV(average values)
0.3	900	0.05	105.4±3
0.3	900	0.1	120±2
0.3	950	0.01	106.6±2
0.3	950	0.1	117.6±3
0.3	1000	0.01	103.7±1
0.3	1000	0.05	98.2±0.8
0.3	1050	0.001	92.6±2.5

4.3.4 GRAPHS

Fig 4.3.4 shows HV plotted against different hot compressed specimens. Here from the graph we can make out that these specimes lying in between 900-950˚C and higher strain rates have higher hardness number and hardness is decreasing as the temperature increases because the material gets more softened

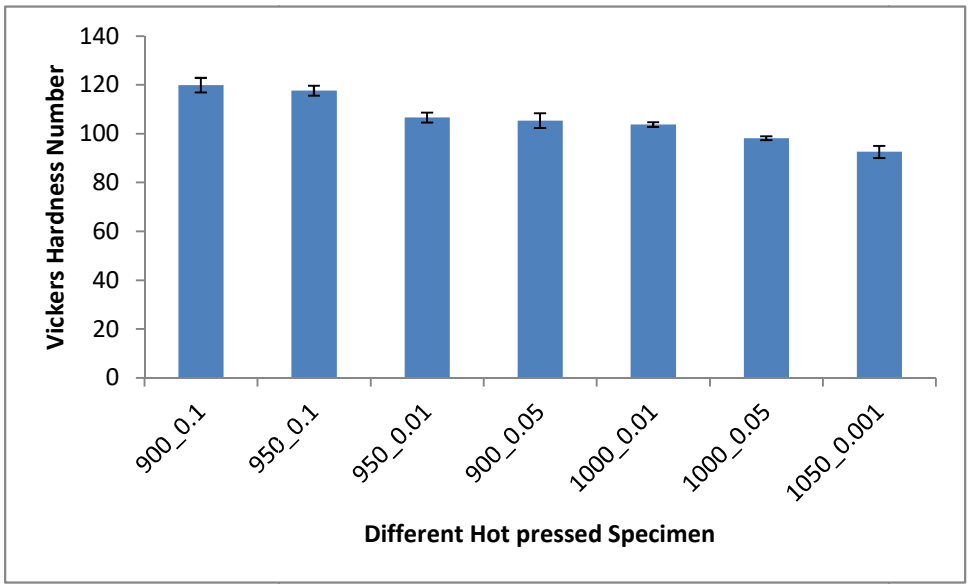


Fig 4.3.4 plot that shows variation of HV with respect different sample

5. CONCLUSIONS

1. Since the as received and solutionised grain size are similar and their Vickers hardness numbers are also similar hence its inferred that as received state of specimen was solutionised.

2. By observing the EDX and SEM photos of Asreceived, solutionised and aged samples . conclusion can be deduced that, Carbides covers the major proportion of the precipates present in specimen, however few borides and silicates are also present and these precipitates are not easily soluble and inorder to find other precipitates more powerful technique needs to be employed.

3. In this case, for Haynes 242; Necklace grains microstructure phenemenon occurs, in those samples which were deformed at very low strain rates and high temperatures, and both the conditions needs to be satisfied. This microstucture takes place between 950 to 1000˚C and further research needs to be done in order to find exact range of temperature.

4. Regarding to the flow curves; apparently, there is no dynamic recrystallization taking place at lower temperatures like 900-950˚C,as there is no significant dip observe on the graph hence it would be recovery whereas at higher temperature(1000˚C-1050˚C) there is Fine recrystallized grain structure called necklace forms along grain boundaries and, to a lesser degree, at twin boundaries.There is no significant dip observe on the graph as these recrystallized structures havent spread across the whole grains and are confined to the grain boundries.

6.BUDGET

The budget for this project will be made from the point of view of an engineering That a company had contracted to do the study. The work includes the cost of Mechanization of the specimens, the microscopic study of the samples obtained, the Treatment and mechanical study of the material.

6.1 COST OF MATERIAL AND HEAT TREATMENT

No cost were incurred as the test material was given by Frisa Superalloy.The heat treatments were also carried out at free of cost.

6.2 COST OF THE MICROSCOPY AND HARDNESS TESTING

The microscopic study of the samples includes the polishing of the samples, and their posterior Analysis with the optical microscope and, for some, with a scanning electron microscope (S.E.M.).The total number of samples studied is 14, of which 4 have been observed in the S.E.M and 7 of them are Hot compressed ones. Thus, the breakdown of the complete microscopic study results is given below:

Table 6.2 Represents various cost incurred due to microscopy and Hardness testing

PROCESS	NUMBER OF SPECIMEN	TOTAL COST(€)
Polishing	14	-
OM(optical microscope)	14	-
SEM (37,8€/hr)	4	151
Hardness testing	10	-
EDX	4	-

Total Cost	151€

6.3 COST OF INSTRON TESTING

Instron Testing was carried on 16 specimens and it costs 160€/test hence, on the whole it costed 2560€.

6.4 TOTAL COST INCURRED DURING EXPERIMENT

The table below comprises of all the cost incurred during the experiment.

Table 6.4 Total summary of all the cost incurred during the experiment

TYPE OF COST INCURRED	PRICE(€)
COST OF MATERIAL AND HEAT TREATMENT	-
COST OF THE MICROSCOPY AND HARDNESS TESTING	151
COST OF INSTRON TESTING	2560

TOTAL	2711€

7. REFERENCES

1. HAYNES® 242® Alloy Brochure - Haynes International.

2. Polish, retrieved 2008-01-04.

3. “Abrasive Grinding Paper”.www.metallographic.com

4. Rajan, T. V.; Sharma, C. P.; Sharma, Ashok (1992). *Heat Treatment: Principles and Techniques*. Prentence Hall. p. 1.

5. Jon L. Dossett.; Howard E. Boyer.Practical Heat Treating: Second Edition

6. "Etching | Definition of etching by Merriam-Webster". Merriam-webster.com. Retrieved 2015-08-11.

7. Peter Farb, Man's Rise to Civilization (1978) p.205, citing Emil Walter Haury, The Hohokam: Desert Farmers and Craftsmen (1976).

8. JR Blueford. "Lesson 2 – Page 3, CLASSIFICATION OF MICROSCOPES". msnucleus.org. Retrieved 15 January 2017.

9. Stokes, Debbie J. (2008). Principles and Practice of Variable Pressure Environmental Scanning Electron Microscopy (VP-ESEM). Chichester: John Wiley & Sons. *ISBN 978-0470758748*.

10. The Vickers Hardness Testing Machine. UKcalibrations.co.uk. Retrieved on 2016-06-03.

11. Joseph Goldstein (2003). Scanning Electron Microscopy and X-Ray Microanalysis. Springer. ISBN 978-0-306-47292-3. Retrieved 26 May 2012.

12. Wai-Fah Chen and Da-Jian Han (2007), "Plasticity for Structural Engineers". J. Ross Publishing ISBN 1-932159-75-4

13. Lubliner, Jacob (2008). Plasticity Theory (PDF) (Revised ed.). Dover Publications. ISBN 0-486-46290-0.

14. Hamed Mirzadeh; Jose Maria Cabrera; Abbas Najafizadeh (2011). Constitutive relationships for hot deformation of austenite.

15. ASM Handbook, Vol. 8, Mechanical Testing and Evaluation, ASM International, Materials Park, OH 44073-0002.

16. http://www.instron.us/en-us/our-company/library/test-types/compression-test

17. https://www.sp.se/en/index/services/rockmechanicaltesting/triaxial/sidor/default.aspx

18. Boresi, A. P, Schmidt, R. J. and Sidebottom, O. M., 1993, Advanced Mechanics of Materials, Wiley.

19.http://www.mechanicalengineeringblog.com/wp-content/uploads/2011/04/01-poissons-ratio-strain-changes.gif

20.http://www.andersonmaterials.com/wp-content/uploads/2013/08/Metallographic-Microscope.jpg

21. http://mcff.mtu.edu/acmal/electronmicroscopy/FE_Form_Function.htm

22. Michael G. Fahrmanna, S. Krishna Srivastava, and Lee M. Pike(2014). HAYNES 244 alloy – a new 760 ◦C capable low thermal expansion alloy

23. S. Dymek, M. Dollar, A. Iyer' and D.L. Klarstrom; EFFECTS OF LONG RANGE ORDERING, TEMPERATURE AND STRAIN RATE ON DEFORMATION BEHAVIOR OF A Ni-Mo-Cr ALLOY

24. https://commons.wikimedia.org/wiki/File:Vickers_anvil_diamons.jpg

25. http://patentimages.storage.googleapis.com/WO1992018660A1/imgf000010_0001.png